EPA Contract No.: 68-W8-0093
Work Assignment No.: 17-5L4J
Donohue Project No.: 20026

VOLUME 3
ADDENDUM TO
FINAL QUALITY ASSURANCE PROJECT PLAN
FOR
HIMCO DUMP
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
PHASE II ACTIVITIES
ELKHART, INDIANA

JULY 1991

Prepared for:

U.S. Environmental Protection Agency
Emergency and Remedial Response Branch
Region V
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DONOHUE & ASSOCIATES, INC.

VOLUME 3
ADDENDUM TO
QUALITY ASSURANCE PROJECT PLAN
HIMCO DUMP RI/FS
PHASE II ACTIVITIES
ELKHART, INDIANA

JULY 1991

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ACRONYMS

Himco Dump QAPP
Elkhart, Indiana

NPL	National Priorities List
OADS	Organic Analysis Data Sheet
OERR	Office of Emergency and Remedial Response, U.S. EPA
PCBs	Polychlorinated Biphenyls
PCB/P	PCBs and Pesticides
pH	Measure of acidity indicated as log of hydrogen ion concentration
PM	Project Manager, Donohue
PRP	Potentially Responsible Party
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAMS	Quality Assurance Management Staff, U.S. EPA
QAPP	Quality Assurance Project Plan
QC	Quality Control
%R	Percent Recovery
RAS	Routine Analytical Services
RI	Remedial Investigation
RMCL	Recommended Maximum Contaminant Level
RPD	Relative Percent Difference
RPM	Remedial Project Manager, U.S. EPA
RPO	Remedial Project Officer, U.S. EPA
RSCC	Regional Sample Control Center, U.S. EPA
SAS	Special Analytical Services
SM	Site Manager, Donohue
SMO	Sample Management Office
SO_4	Sulfate
SOP	Standard Operating Procedure
SOW	Statement of Work
SQCO	Site Quality Control Officer, Donohue
TAC	Technical Advisory Committee, Donohue
TAL	Target Analyte List
TCL	Target Compound List
TDS	Total Dissolved Solids
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
TP	Total Phosphorus
TSQAM	Technical Services Quality Assurance Manager, Donohue
TSS	Total Suspended Solids
USGS	United States Geological Survey
VOA	Volatile Organic Compounds
VOC	Volatile Organic Compounds (same as VOA)

ACRONYMS

Himco Dump QAPP
Elkhart, Indiana

ABN	Acid and Base-Neutral Semivolatile Organic Compounds
ASTM	American Society of Testing Materials
BNA	Base-Neutral and Acid Semivolatile Organic Compounds (same as ABN)
BOD	Biochemical Oxygen Demand
CDO	Central District Office, U.S. EPA Region V
CH_4	Methane
Cl	Chloride
CLP	Contract Laboratory Program
CN	Cyanide
COD	Chemical Oxygen Demand
COE	Corps of Engineers, U.S. Army
CRDL	Contract Required Detection Limit
CRL	Central Regional Laboratory, U.S. EPA Region V
CRQL	Contract Required Quantitation Limit
DO	Dissolved Oxygen
DQO	Data Quality Objective
E&E	Ecology & Environment, Inc.
EM	Electromagnetic Meter
EMSL	Environmental Monitoring and Support Laboratory, U.S. EPA
EPA	U.S. Environmental Protection Agency
FIT	Field Investigation Team
FS	Feasibility Study
FSP	Field Sampling Plan
FTL	Field Team Leader, Donohue
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
HNO_3	Nitric Acid
HNu	Photoionization detector manufacturer
HQ	Headquarters, U.S. EPA
H_2S	Hydrogen Sulfide
IADS	Inorganic Analysis Data Sheet
ICP	Inductively Coupled Argon Plasma Spectrometer
I.D.	Inner Diameter
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
ISBH	Indiana State Board of Health
LSSS	Laboratory Support Services Section, U.S. EPA Region V
Lumidor	Gas monitoring device manufacturer
MCL	Maximum Contaminant Level
NC	Not Calculated
NEIC	National Enforcement Investigations Center, U.S. EPA
NH_3	Ammonia Nitrogen
NO_2+NO_3	Nitrite + Nitrate Nitrogen

- Chemical and geotechnical analysis of soil borings.
- Sampling and chemical analysis of new wells and existing U.S.G.S. wells.
- Residential well sampling and chemical analysis.
- Collection of waste mass gas samples from the fill for analysis of volatile organics.
- Residential basement air sampling and analysis for methane, hydrogen sulfide, and volatile organics.
- Collection of landfill cap samples for analysis of geotechnical properties.
- Sampling and chemical analysis of landfill cap surface.
- Sampling and chemical analysis of sediment and surface water samples from three on-site water bodies.
- Installation of staff gauges, measurement of water levels, and slug tests to determine site hydrology.

3.4 PROJECT OBJECTIVES

3.4.1 Objectives

Following the review of Phase I sampling results, additional data were identified which are necessary to complete the risk assessment and feasibility study. The objectives of the Phase II sampling and analysis program are discussed below.

3.4.1.1 Private Well Inventory

Phase I groundwater sampling detected contaminants at values which were not high enough to be of concern from a risk assessment standpoint, however, several contaminants exceeded established MCLs. Contaminants were found in downgradient wells screened from 15 to 175 feet, but very near the landfill, yet vertical downward gradients are near nonexistent. It is hypothesized that the pumping of private wells in the area had a significant effect on the groundwater flow near the site. Because of the influence of pumping wells on the local flow pattern, an assessment of the screened depths and lengths, and pumping rates of all private wells in the vicinity of the site will be performed.

3.4.1.2 Trenching for Leachate Sampling and Debris Delineation

Contaminated groundwater (leachate) was observed draining from pockets of waste debris within the calcium sulfate matrix. Samples of this leachate will be collected by re-excavating previous trench locations and dipping a sample collection jar into the leachate that collects in the bottom of the trench.

3.0 PROJECT DESCRIPTION

3.1 INTRODUCTION

Donohue & Associates, Inc., (Donohue) is submitting this addendum to the approved Final Quality Assurance Project Plan (QAPP) for the Himco Dump Superfund Site Remedial Investigation/Feasibility Study (RI/FS) (June 1990) to address the data quality for the Phase II RI. This document is Volume 3 of the Himco Dump Work Plan Addendum. The purpose of the Phase II RI is to fill the data needs identified for the risk assessment, ecological assessment, and feasibility study.

The Phase II investigation will include surface water, leachate, sediment and soil sampling, and chemical analysis. A well inventory will be conducted. The wetland south of the quarry will be delineated based on vegetation and soil types. The wetland remnant area which contained debris and polynuclear aromatics (PNA) will be delineated using trenches. The landfill cap will be sampled for the geotechnical property of triaxial shear. Benthic invertebrates will be collected and identified. Mammal populations will be estimated based on tracks and scat.

This QAPP addendum supplements the June 1990 approved Final QAPP and addresses only those new activities to be conducted during Phase II. Reference to the sections in the QAPP plan which apply are also included.

3.2 SITE DESCRIPTION

The site description contained in Section 3.2 of the approved QAPP applies.

3.3 SITE HISTORY AND BACKGROUND

The site history and description contained in Section 3.3 of the approved QAPP applies.

In October 1990 through January 1991, a Phase I RI was conducted by Donohue. The RI consisted of:

- Electromagnetic (EM) and magnetometer survey to determine boundaries of the fill and presence of metal/drums in the fill.
- Excavation of test pits to determine source of em and magnetic anomalies found during the geophysical survey.
- Preliminary assessment of potential wetland areas by vegetation and soil type.
- Sampling and chemical analysis of soil from potential wetland areas.
- Installation of ten new monitoring wells at soil boring locations.

3.4.1.5 Landfill Cap Soil Sampling for Geotechnical Analysis

Five soil samples will be collected of the landfill cap using a hand auger. These disturbed samples will be shipped to a geotechnical lab for triaxial shear tests in order to evaluate landfill capping options. Previous samples collected for these tests did not provide all of the required data. Additional samples are required to account for the soil variability observed in the existing landfill cover.

3.4.2 Data Uses

The data collected during the Phase II RI will be used to satisfy data needs associated with site characterization, risk assessment, ecological assessment, and evaluation of remedial alternatives during the FS. Data needs specific to the Himco Dump RI/FS have been identified by evaluating existing data with reference to the Conceptual Site Model and are discussed in Section 4.0 of the Work Plan Addendum (Volume 1A).

Users of the data generated from the Phase II RI will consist of the EPA, IDEM, health assessment scientists, engineers, hydrogeologists, geologists, mammalogists, BTAG, biologists, chemists, potentially responsible parties (PRPs), the U.S. Fish and Wildlife Service, and the Corps of Engineers.

3.5 CONTAMINANTS OF CONCERN AND POTENTIAL SOURCE AREAS

The DQO levels proposed for Phase II are listed in Table 3-4. DQO summary forms used during the scoping process are included in Appendix B.

Level I Field Screening/Real Time

Benthos (macroinvertebrate) identification
Depth of soil and sediment organic layer
pH, conductivity, dissolved oxygen, and temperature of surface water and leachate
VOA by HNu, methane and hydrogen sulfide for ambient air monitoring during trenching

Level IV CLP Routine Analytical Services

TCL VOA in surface water, leachate, soil, and sediment
TCL BNA in surface water, leachate, soil, and sediment
TCL PCB/Pesticides in surface water, leachate, soil, and sediment
TAC Metals/CN in surface water, leachate, soil, and sediment

Level V CLP SAS

Triaxial shear of landfill cap soil
Grain size of soil
TOC in soil and sediment
Water quality of surface water and leachate

The leachate samples will be analyzed to provide data to be used for determining remediation methods, and for providing data to the POTW for pretreatment assessment.

In addition, up to 20 trenches will be excavated to delineate the thickness and lateral extent of construction debris associated with high PAH values detected in soil samples taken during Phase I wetland soil sampling.

3.4.1.3 Surface Water and Sediment

Two sample locations at the "L" shaped pond, one at the small fish pond and three at the quarry pond will be sampled for surface water and sediment. Samples will be taken from deeper water near the pond centers. A temperature probe will be lowered to the bottom to develop a temperature profile of each pond. A dredge sampler will be used for gathering sediment for analysis and benthic organisms. A gravity core device will be used to collect a sediment profile of the lake bottom and to provide sediment for geotechnical analysis. Surface water will be collected for analysis at the same locations as sediment samples.

3.4.1.4 Wetland and Other Surface Soil Sampling

A preliminary wetland delineation was performed during Phase I activities. True wetlands were not identified other than an area south of the quarry pond. A second delineation will include a refined wetland boundary determination of this area, and the collection of soil samples for chemical analysis.

A surface water drainage study performed by Donohue showed that one of the major directions of surface water drainage is west off of the landfill. In order to investigate the potential impact to surface soils due to surface water draining off of the landfill towards the fish ponds, other surface soil samples for chemical analysis will be collected west of the landfill cap between the landfill and fish ponds.

A dirt bike and foot trail has developed along the south quarry pond fence by trespassers. Three surface soil samples will be collected along this path to investigate for contamination which could potentially effect the trail users.

TABLE 3-4

SAMPLING AND ANALYSIS SUMMARY TABLE FOR HIMCO DUMP RI/FS

PHASE II (Continued)

							FIELD QC						LAB QC			
SAMPLE MATRIX	FIELD PARAMETERS	DQO LEVEL	LAB PARAMETERS	DQO LEVEL	LAB	FIELD SAMPLES	BB(1)	BG	TB(2)	FB	FD	TOTAL TO LAB	LD	MSD(3)	MS	PURPOSE OF SAMPLES
SURFACE WATER (QUARRY, 2 FISH PONDS)	pH	I	TCL VOA	IV	CLP	12 Max	-	3	1	2	2	20	-	1	1	Ecological and human risk assessment
	Conductivity	I	TCL BNA	IV	CLP	9 Max	-	3	-	2	2	16	-	1	1	
	DO	I	TCL PCB/P	IV	CLP	9 Max	-	3	-	2	2	16	-	1	1	
	Temperature	I	Total Metals-Total & Dissolved (4)/CN	IV	CLP	9 Max	-	3	-	2	2	16	1	-	1	
			Water Quality (5)	V	CLP SAS	9 Max	-	3	-	2	2	16	1	-	1	
			COD	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			Cl	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			SO4	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			NH3	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			NO2 + NO3	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			TKN	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			TP	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			TDS	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	-	
			TSS	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	-	
			alkalinity	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	
			Bromide (Diss)	V	CLP SAS	9 Max	-	3	-	2	2	16	2	-	2	

NOTES:

1 Assume bottle cleaning protocol submitted with QAPP acceptable.

2 Trip blanks will be shipped at a frequency of one per cooler of aqueous samples for VOA analysis.

3 MS/MSD samples required for organic analysis. Aqueous samples (surface water, leachate) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

4 Total metals are defined as digestion and analysis of TAL metals on an unfiltered sample. Dissolved metals are defined as digestion and analysis of TAL metals on a sample filtered in the field.

5 Water quality analyses will be done on unfiltered sample except for bromide which will be field filtered.

LEGEND

BB = Bottle Blank
BG = Background Sample
TB = Trip Blank
FB = Field Blank
FD = Field Duplicate
LD = Lab Duplicate
MSD = Matrix Spike Duplicate
MS = Matrix Spike
- = Not Applicable

Diss = Dissolved

TABLE 3-4

SAMPLING AND ANALYSIS SUMMARY TABLE FOR HIMCO DUMP RI/FS

PHASE II

							FIELD QC						LAB QC			
SAMPLE MATRIX	FIELD PARAMETERS	DQO LEVEL	LAB PARAMETERS	DQO LEVEL	LAB	FIELD SAMPLES	BB(1)	BG	TB(2)	FB	FD	TOTAL TO LAB	LD	MSD(3)	MS	PURPOSE OF SAMPLES
LEACHATE	pH	I	TCL VOA	IV	CLP	7 Max	-	-	1	1	1	10	-	1	1	Waste characterization of primary source
	Conductivity	I	TCL BNA	IV	CLP	7 Max	-	-	-	1	1	9	-	1	1	Risk assessment
	DO	I	TCL PCB/P	IV	CLP	7 Max	-	-	-	1	1	9	-	1	1	Evaluate remedial alternatives
	Temperature	I	Total Metals (4)/CN	IV	CLP	7 Max	-	-	-	1	1	9	1	-	1	
			Water Quality	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			COD	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			Cl	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			SO4	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			NH3	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			NO2 + NO3	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			TKN	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			TP	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			TDS	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			TSS	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			alkalinity	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			BOD	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			VSS	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			O and G	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			Total Phenol	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	
			Bromide (Diss)	V	CLP SAS	7 Max	-	-	-	1	1	9	1	-	1	

NOTES:

1 Assume bottle cleaning protocol submitted with QAPP acceptable.

2 Trip blanks will be shipped at a frequency of one per cooler of aqueous samples for VOA analysis.

3 MS/MSD samples required for organic analysis. Aqueous samples (surface water, leachate) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

4 Total metals are defined as digestion and analysis of TAL metals on unfiltered samples.

LEGEND

BB = Bottle Blank
BG = Background Sample
TB = Trip Blank
FB = Field Blank
FD = Field Duplicate
LD = Lab Duplicate
MSD = Matrix Spike Duplicate
MS = Matrix Spike
- = Not Applicable
Diss = Dissolved

TABLE 3-4

SAMPLING AND ANALYSIS SUMMARY TABLE FOR HIMCO DUMP RI/FS

PHASE II (Continued)

							FIELD QC						LAB QC			
SAMPLE MATRIX	FIELD PARAMETERS	DQO LEVEL	LAB PARAMETERS	DQO LEVEL	LAB	NUMBER OF FIELD SAMPLES	BB(1)	BG	TB(5)	FB	FD	TOTAL TO LAB	LD	MSD(4)	MS	PURPOSE OF SAMPLES
EXISTING AND NEW WELLS GROUNDWATER	Water Level	I	TCL VOA	IV	CLP	18	-	-	2	2	2	24	-	2	2	Determine nature and extent of contamination
	pH	I	TCL BNA	IV	CLP	18	-	-	-	2	2	22	-	2	2	
	Conductivity	I	TCL PCB/P	IV	CLP	18	-	-	-	2	2	22	-	2	2	
	DO	I	Total Metals/CN	IV	CLP	18	-	-	-	2	2	22	2	-	2	Evaluate remedial alternatives
	Temperature	I,	(Total and Dissolved) (2)													Risk assessment
			Water Quality (3)	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			COD	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			Cl	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			SO4	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			NH3	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			NO2 + NO3	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			TKN	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			TP	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			TDS	V	CLP SAS	18	-	-	-	2	2	22	3	-	-	
			TSS	V	CLP SAS	18	-	-	-	2	2	22	3	-	-	
			alkalinity	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
			bromide, dissolved	V	CLP SAS	18	-	-	-	2	2	22	3	-	3	
SOIL FROM NEW WELL INSTALLATION	VOAs by HNu	I	TCL VOA	IV	CLP	4 Max	-	-	-	-	1	5	-	-	-	Determine subsurface soil chemistry for evaluation of remedial alternatives nature and extent of contamination
			TCL BNA	IV	CLP	4 Max	-	-	-	-	1	5	-	-	-	
			TCL PCB/P	IV	CLP	4 Max	-	-	-	-	1	5	-	-	-	
			Total Metals/CN	IV	CLP	4 Max	-	-	-	-	1	5	-	-	-	
			TOC	V	CLP SAS	4 Max	-	-	-	-	1	5	-	-	-	
			Grain Size	V	CLP SAS	4 Max	-	-	-	-	1	5	-	-	-	

NOTES:

1 Assume bottle cleaning protocol submitted with QAPP acceptable.

2 Total metals are defined as digestion and analysis of TAL metals on an unfiltered sample. Dissolved metals are defined as digestion and analysis of TAL metals on a sample filtered in the field.

3 Water quality analysis will be done on unfiltered sample except for bromide which will be field filtered.

4 MS/MSD samples required for organic analysis. Aqueous samples (groundwater, surface water, leachate, priavate well water) shall be collected, with extra sample volume at a frequency of one per 20 fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

5 Trip blanks will be shipped at a frequency of one per cooler of aqueous samples for VOA analysis.

LEGEND

BB = Bottle Blank
BG = Background Sample
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FB = Field Blank
FD = Field Duplicate
LD = Lab Duplicate
MSD = Matrix Spike Duplicate
MS = Matrix Spike
- = Not Applicable

TABLE 3-4

SAMPLING AND ANALYSIS SUMMARY TABLE FOR HIMCO DUMP RI/FS

PHASE II (Continued)

							FIELD QC						LAB QC			
SAMPLE MATRIX	FIELD PARAMETERS	DQO LEVEL	LAB PARAMETERS	DQO LEVEL	LAB	FIELD SAMPLES	BB(1)	BG	TB(2)	FB	FD	TOTAL TO LAB	LD	MSD(3)	MS	PURPOSE OF SAMPLES
SEDIMENT (QUARRY, FISH PONDS)	Benthos ID	I	TCL VOA	IV	CLP	6 Max	-	3	-	-	2	11	-	1	1	Ecological and human risk assessment
		I	TCL BNA	IV	CLP	6 Max	-	3	-	-	2	11	-	1	1	
		I	TCL PCB/P	IV	CLP	6 Max	-	3	-	-	2	11	-	1	1	
		I	Total Metals/CN	IV	CLP	6 Max	-	3	-	-	2	11	1	-	1	
	Depth of organic layer	I	TOC	V	CLP SAS	6 Max		3			2	11	1	-	-	
			Grain Size	V	CLP SAS	6 Max		3			2	11	1	-	-	
WETLAND SOIL BIKE TRAIL RUNOFF SOIL SOILS	Depth of organic layer	I	TCL VOA	IV	CLP	8 Max	-	-	-	-	1	9	-	1	1	Ecological and human risk assessment
			TCL BNA	IV	CLP	8 Max	-	-	-	-	1	9	-	1	1	
			TCL PCB/P	IV	CLP	8 Max	-	-	-	-	1	9	-	1	1	
			Total Metals/CN	IV	CLP	8 Max	-	-	-	-	1	9	1	-	1	
			TOC	V	CLP SAS	8 Max	-	-	-	-	1	9	1	-	-	
			Grain Size	V	CLP SAS	8 Max	-	-	-	-	1	8	-	-	-	
LANDFILL CAP SOIL			Triaxial Compression	V	CLP SAS	5 Max	-	-	-	-	-	5	-	-	-	Evaluate remedial alternatives

NOTES:

1 Assume bottle cleaning protocol submitted with QAPP acceptable.

2 Trip blanks will be shipped at a frequency of one per cooler of aqueous samples for VOA analysis.

3 MS/MSD samples required for organic analysis. Aqueous samples (surface water, leachate) shall be collected, with extra sample volume at a frequency of one per 20 or fewer investigative samples. Triple the normal volume will be collected for VOAs and double the normal volume will be collected for BNA and PCB/P.

LEGEND

BB = Bottle Blank
BG = Background Sample
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LD = Lab Duplicate
MSD = Matrix Spike Duplicate
MS = Matrix Spike
- = Not Applicable
Diss = Dissolved

Analysis of Phase II site samples for the water quality and geotechnical parameters will be accomplished through the CLP Special Analytical Services (SAS) program. Detection limits for SAS analysis are included in the specific SAS requests in Appendix A. Quantitation limits for TAL metals/cyanide and TCL volatile organics, semivolatile organics, and PCBs/pesticides are contained in Tables 3-1 and 3-2. Quantitation limits for SAS analytes are contained in Table 3-3.

The contaminants of concern, sampling media and sampling locations for Phase II were selected based on the chemical results obtained during Phase I. A Phase II RI scoping meeting was held with the U.S. EPA RPM, Indiana Department of Environmental Management (IDEM), Donohue, and risk assessment, ecological assessment, and air pathway scientists. The following matrices and analytes were decided on:

3.5.1 Leachate

Leachate seeps were not present during the Phase I RI field investigation. Test pits excavated to investigate em and magnetic anomalies quickly filled with dark colored liquid which exhibited HNu readings exceeding 5 ppm. This trench liquid (leachate) was not sampled during the Phase I RI. Characterization of leachate is necessary for assessment of pump and treat remedial alternatives for restoration of the aquifer if it is determined to be a sole source aquifer. Residential wells located immediately downgradient of the site are believed to be impacted by the leachate as evidenced by high COD, NH_3, and TKN. Bromide was detected in residential and groundwater monitoring wells located downgradient of the site. Cadmium, chromium, lead, and benzene exceeding the MCLs were also detected downgradient. Phenobarbital, identified by the GC/MS computer library search, was also present in adjacent residential wells.

Accordingly, leachate will be collected from three trenches dug on-site with a backhoe and analyzed for TCL VOA, TCL BNA, TCL PCB/Pesticides, and TAL metals/CN to characterize the contaminant source. Analytes for the assessment of leachate treatment alternatives are:

Biochemical Oxygen Demand (BOD): Measure oxygen load of waste to treatment technology.

Chemical Oxygen Demand (COD): Measure of chemical oxidation in water; poorly degradable contaminants will elevate COD above BOD level.

Chloride: Major mobile anion associated with typical landfill leachate.

Sulfate: Anion associated with typical landfill leachate; reduction of organic sulfur occurs in anaerobic conditions and oxidation occurs in aerobic conditions.

Ammonia, Nitrate and Nitrite, Total Kjeldahl Nitrogen: Speciation of nitrogen needed for remedial alternative selection.

TABLE 3-1

QUANTITATION LIMITS FOR RAS TAL COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana
(Continued)

For lead:

Method in use - ICP
Instrument Detection Limit (IDL) - 40
Sample concentration - 220
Contract Required Detection Limit (CRDL) = 3

The value of 220 may be reported even though instrument detection limit is greater than CRDL. The instrument or method detection limit must be documented as described in Exhibit E.

2. The CRDL are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E. The detection limits for samples may be considerably higher depending on the sample matrix.

3. The detection limits for soil samples are approximately 200 times those for water samples (per Region V Sample Handling Manual, March, 1989).

TABLE 3-1

QUANTITATION LIMITS FOR RAS TAL COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

Analyte	Contract Required Detection Limit (CRDL) Water (ug/L) (1,2)	Soil (mg/kg) (3)
Aluminum	200	40
Antimony	60	12
Arsenic	10	2
Barium	200	40
Beryllium	5	1
Cadmium	5	1
Calcium	5000	1000
Chromium	10	2
Cobalt	50	10
Copper	25	5
Iron	100	20
Lead	3	1
Magnesium	5000	1000
Manganese	15	3
Mercury	0.2	0.040
Nickel	40	8
Potassium	5000	1000
Selenium	5	1
Silver	10	2
Sodium	5000	1000
Thallium	10	2
Vanadium	50	10
Zinc	20	4
Cyanide	10	2

1. Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods - Catastrophic Failure) any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

   If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

TABLE 3-2
(continued)

QUANTITATION LIMITS FOR RAS TCL ORGANIC COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

Semi-volatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
34. Phenol	108-95-2	10	330	10000	(20)
35. bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
36. 2-Chlorophenol	95-57-8	10	330	10000	(20)
37. 1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)
38. 1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)
39. 1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)
40. 2-Methylphenol	95-48-7	10	330	10000	(20)
41. 2,2'-oxybis (1-Chloropropane)**	108-60-1	10	330	10000	(20)
42. 4-Methylphenol	106-44-5	10	330	10000	(20)
43. N-Nitroso-di-n dipropylamine	621-64-7	10	330	10000	(20)
44. Hexachloroethane	67-72-1	10	330	10000	(20)
45. Nitrobenzene	98-95-3	10	330	10000	(20)
46. Isophorone	78-59-1	10	330	10000	(20)
47. 2-Nitrophenol	88-75-5	10	330	10000	(20)
48. 2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
49. bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)
50. 2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
51. 1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)
52. Naphthalene	91-20-3	10	330	10000	(20)
53. 4-Chloroaniline	106-47-8	10	330	10000	(20)
54. Hexachlorobutadiene	87-68-3	10	330	10000	(20)
55. 4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
56. 2-Methylnaphthalene	91-57-6	10	330	10000	(20)
57. Hexachlorocyclopentadiene	77-47-4	10	330	10000	(20)
58. 2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
59. 2,4,5-Trichlorophenol	95-95-4	25	800	25000	(50)
60. 2-Chloronaphthalene	91-58-7	10	330	10000	(20)
61. 2-Nitroaniline	88-74-4	25	800	25000	(50)
62. Dimethylphthalate	131-11-3	10	330	10000	(20)
63. Acenaphthylene	208-96-8	10	330	10000	(20)
64. 2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65. 3-Nitroaniline	99-09-2	25	800	25000	(50)
66. Acenaphthene	83-32-9	10	330	10000	(20)
67. 2,4-Dinitrophenol	51-28-5	25	800	25000	(50)

TABLE 3-2
(continued)

QUANTITATION LIMITS FOR RAS TCL ORGANIC COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

		Quantitation Limits*			
Semi-volatiles	CAS Number	Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
68. 4-Nitrophenol	100-02-7	25	800	25000	(50)
69. Dibenzofuran	132-64-9	10	330	10000	(20)
70. 2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71. Diethylphthalate	84-66-2	10	330	10000	(20)
72. 4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73. Fluorene	86-73-7	10	330	10000	(20)
74. 4-Nitroaniline	100-01-6	25	800	25000	(50)
75. 4,6-Dinitro-2-methylphenol	534-52-1	25	800	25000	(50)
76. N-nitrosodiphenylamine	86-30-6	10	330	10000	(20)
77. 4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78. Hexachlorobenzene	118-74-1	10	330	10000	(20)
79. Pentachlorophenol	87-86-5	25	800	25000	(50)
80. Phenanthrene	85-01-8	10	330	10000	(20)
81. Anthracene	120-12-7	10	330	10000	(20)
82. Carbazole	86-74-8	10	330	10000	(20)
83. Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84. Fluoranthene	206-44-0	10	330	10000	(20)
85. Pyrene	129-00-0	10	330	10000	(20)
86. Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87. 3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88. Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89. Chrysene	218-01-9	10	330	10000	(20)
90. bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)
91. Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92. Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93. Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94. Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95. Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)
96. Dibenz(a,h)anthracene	53-70-3	10	330	10000	(20)
97. Benzo (g,h,i)perylene	191-24-2	10	330	10000	(20)

TABLE 3-2
QUANTITATION LIMITS FOR RAS TCL ORGANIC COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

Volatiles	CAS Number	Quantitation Limits* Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
1. Chloromethane	74-87-3	10	10	1200	(50)
2. Bromomethane	74-83-9	10	10	1200	(50)
3. Vinyl Chloride	75-01-4	10	10	1200	(50)
4. Chloroethane	75-00-3	10	10	1200	(50)
5. Methylene Chloride	75-09-2	10	10	1200	(50)
6. Acetone	67-64-1	10	10	1200	(50)
7. Carbon Disulfide	75-15-0	10	10	1200	(50)
8. 1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9. 1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11. Chloroform	67-66-3	10	10	1200	(50)
12. 1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13. 2-Butanone	78-93-3	10	10	1200	(50)
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15. Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16. Bromodichloromethane	75-27-4	10	10	1200	(50)
17. 1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18. cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19. Trichloroethene	79-01-6	10	10	1200	(50)
20. Dibromochloromethane	124-48-1	10	10	1200	(50)
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22. Benzene	71-43-2	10	10	1200	(50)
23. trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24. Bromoform	75-25-2	10	10	1200	(50)
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26. 2-Hexanone	591-78-6	10	10	1200	(50)
27. Tetrachloroethene	127-18-4	10	10	1200	(50)
28. Toluene	108-88-3	10	10	1200	(50)
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30. Chlorobenzene	108-90-7	10	10	1200	(50)
31. Ethyl Benzene	100-41-4	10	10	1200	(50)
32. Styrene	100-42-5	10	10	1200	(50)
33. Xylenes (Total)	1330-20-7	10	10	1200	(50)

TABLE 3-2
(continued)

QUANTITATION LIMITS FOR RAS TCL ORGANIC COMPOUNDS
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

Pesticides/Aroclors	CAS Number	Quantitation Limits* Water ug/L	Soil ug/Kg	On Column (pg)
98. alpha-BHC	319-84-6	0.05	1.7	5
99. beta-BHC	319-85-7	0.05	1.7	5
100. delta-BHC	319-86-8	0.05	1.7	5
101. gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
102. Heptachlor	76-44-8	0.05	1.7	5
103. Aldrin	309-00-2	0.05	1.7	5
104. Heptachlor epoxide	1024-57-3	0.05	1.7	5
105. Endosulfan I	959-98-8	0.05	1.7	5
106. Dieldrin	60-57-1	0.10	3.3	10
107. 4,4'-DDE	72-55-9	0.10	3.3	10
108. Endrin	72-20-8	0.10	3.3	10
109. Endosulfan II	33213-65-9	0.10	3.3	10
110. 4,4'-DDD	72-54-8	0.10	3.3	10
111. Endosulfan sulfate	1031-07-8	0.10	3.3	10
112. 4,4'-DDT	50-29-3	0.10	3.3	10
113. Methoxychlor	72-43-5	0.50	17.0	50
114. Endrin ketone	53494-70-5	0.10	3.3	10
115. Endrin aldehyde	7421-36-3	0.10	3.3	10
116. alpha-Chlordane	5103-71-9	0.05	1.7	5
117. gamma-Chlordane	5103-74-2	0.05	1.7	5
118. Toxaphene	8001-35-2	5.0	170.0	500
119. Aroclor-1016	12674-11-2	1.0	33.0	100
120. Aroclor-1221	11104-28-2	2.0	67.0	200
121. Aroclor-1232	11141-16-5	1.0	33.0	100
122. Aroclor-1242	53469-21-9	1.0	33.0	100
123. Aroclor-1248	12672-29-6	1.0	33.0	100
124. Aroclor-1254	11097-69-1	1.0	33.0	100
125. Aroclor-1260	11096-82-5	1.0	33.0	100

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

** Previously known by the name bis(2-Chloroisopropyl) ether.

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Pesticides/Aroclors.

Total Phosphorus: Indicator of agricultural use (on-site cropland); high levels enhance algae growth and may complicate some treatment alternatives.

Total Dissolved Solids, Total Suspended Solids: Remedial alternatives such as filtration and sorption are dependent on solids loading.

Alkalinity: Measure of buffering effect and ability to support algae growth; high levels may complicate some treatment alternatives.

Volatile Suspended Solids: Provides rough approximation of organic carbon associated with influent to treatment technology.

Oil and Grease: To assess amount present as excessive levels cause scum accumulation in digesters, clog filter pores, and quickly foul activated carbon.

Total Phenol: Assess if phenol removal is necessary prior to chlorination treatment.

3.5.2 Surface Water

All three surface water bodies on-site were sampled from the shoreline during Phase I. The center of the ponds and quarry will be sampled in Phase II to characterize the surface water where impacted biota may reside. Additionally, a background upgradient surface water body will be sampled. Phase I RI data indicated lead in all three surface water bodies exceeded the Ambient Water Quality Criteria (AWQC). The quarry also contained chromium in exceedance of the AWQC. Concentrations of aluminum and iron in the smallest pond exceeded the Secondary Maximum Contaminant Level (SMCL).

Although no detectable VOA, BNA, PCB/Pesticides or bromide were present in shoreline samples, these analytes will again be measured for in Phase II samples to fully characterize the surface water quality. The water quality parameters of COD, Cl, SO_4, NH_3, TKN, TP, TDS, TSS, and alkalinity measured in Phase I samples will also be included in Phase II. Thermal stratification in the quarry will be determined to assess if both epilimnion and hypolimnion will need to be sampled.

3.5.3 Sediment

All three surface water bodies on-site were sampled from the shoreline for sediment during Phase I. Sediment from the center of the ponds and quarry will be sampled in Phase II to characterize the sediment where impacted biota may reside or feed. Identification of macroinvertebrates in site sediments will be done in the field to determine if species indicative of polluted/ stressed waters are present. Additionally, a background upgradient sediment location will be sampled for comparison. TOC and depth of the sediment layer will be measured for assessment of contaminant sorption characteristics and volume of sediment that may need to be remediated.

The proposed schedule assumes ready access to the site. The proposed schedule also assumes that health and safety personnel protection requirements are Level D, with possible upgrade to Level C, as detailed in the Himco Dump RI/FS Health and Safety Plan (Volume 4). Level B protection will be required during trenching and test pit excavations and leachate sampling. Variation from these assumptions may impact the schedule.

TABLE 3-3

QUANTITATION LIMITS FOR SAS ANALYTES
PHASE II HIMCO DUMP RI/FS
Elkhart, Indiana

SAS Analyte	Matrix	Quantitation Limit
Chloride	Leachate, Surface Water	5 mg/L
Sulfate	Leachate, Surface Water	5 mg/L
TDS	Leachate, Surface Water	20 mg/L
TSS	Leachate, Surface Water	2-3 mg/L
Alkalinity	Leachate, Surface Water	2 mg/L
TP	Leachate, Surface Water	0.05 mg/L
TKN	Leachate, Surface Water	0.1 mg/L
NH_3	Leachate, Surface Water	0.1 mg/L
NO_2+NO_3	Leachate, Surface Water	0.10 mg/L
COD	Leachate, Surface Water	5 mg/L
Bromide	Leachate, Surface Water	0.10 mg/L
Oil & Grease	Leachate Only	0.4 mg/L
VSS	Leachate Only	4 mg/L
Total Phenol	Leachate Only	2 ug/L
BOD	Leachate Only	2 mg/L
TOC	Soil/Sediment	0.10%
Grain Size	Soil/Sediment	1%
Triaxial Shear	Soil	Not Applicable

A/PROJ/HIMCO/AH0

Phase I RI data indicated trace levels (1 ug/kg) of trichloroethylene, tetrachloroethene, trichloroethane, chloroform, and xylene in site sediments. Benzoic acid was also detected at levels of 93-190 ug/kg in the quarry. Aroclor 1248 (130 ug/kg) was reported in one sample out of four collected in the fish pond. Therefore, for Phase II all of these analytes will be measured. Although metal levels were not high enough to classify the sediments as hazardous waste, metals will be measured during Phase II to assess if the chemistry of the sediments in the center of the water bodies differs from the shore and could contribute to the elevated lead and chromium detected in Phase I surface water samples.

3.5.4 Soil

Three possibly impacted areas will be sampled and chemically analyzed during Phase II to fill data needs for the risk and ecological assessments. The wetland area south of the quarry, soil west of the fill located in the drainage path from the site, and surface soil in the exposed area used as a dirt bike trail south of the quarry will be sampled. These areas are discussed in the following paragraphs.

3.5.4.1 Wetland

The only area identified as a true wetland during Phase 1 is located south of the quarry. The surficial soil samples were collected in this area during Phase I. This wetland area will be investigated during Phase II and the boundaries determined through identification of vegetation and soil types. The depth of the soil organic layer and TOC in the wetland will be measured to determine the potential for contaminant attenuation. Grain size will be measured to assess migration of wetland soil. Three surficial soil samples will be collected within the boundaries of the wetland to assess if it is impacted from the site and poses a threat to wetland vegetation and wildlife. The National Wetlands Inventory will be consulted to see if this area is included, and a botanical inventory will be conducted. Identification of endangered species native to the area will be performed prior to wetlands delineation. Chemical analysis will include VOC, BNA, PCB/Pesticides, and metals to characterize impacts from the site.

3.5.4.2 Runoff

A drainage analysis indicates the potential for surface runoff to the west towards Manning Ditch. Two surficial soil samples will be collected in the identified drainageway and analyzed for the parameters listed in Section 3.5.4.1 to determine contaminant migration.

3.5.4.3 Bike Trail

Trespassers appear to ride dirt bikes along the southern and eastern edge of the quarry. Exposed soil and calcium sulfate was noted in these areas but no Phase I samples were not collected. Dust impacts to exposed bike riders will be modelled using data collected during Phase I for landfill cap soil. The chemical results from the surface soil samples collected in this area during Phase II will be used to verify the model. Samples will be analyzed for the parameters listed in Section 3.5.4.1 to determine if exposure could occur.

3.5.5 Landfill Cap

Geotechnical samples collected during the Phase I RI did not yield enough useful data to assess if the landfill can support a structure or a cap. A geotechnical engineer will do a site walk-through during Phase II and identify the type of foundation and surficial soils. He will also locate sampling locations for triaxial shear tests to determine the angle of friction between the existing cap and any proposed cap.

3.5.6 Debris Area

Phase I soil collected in the wetland remnant area located south of the fill contained detectable levels (6-23 ppm) of polynuclear aromatics (PNAs), lead, copper, and cyanide. This area was wetland prior to 1973. Debris was dumped in this area starting in 1973. In order to determine the area and volume of contaminated soil possibly needing remediation, backhoe pits will be excavated to define the extent and type of debris. One of the three leachate samples in 3.4.1 will be collected from a trench dug in the wetland remnant area.

3.6 SAMPLE NETWORK DESIGN AND RATIONALE

A sampling and analysis summary table is presented in Table 3-4. Complete descriptions of sampling activities including sampling location diagrams, sample numbers, and rationale for selected locations are presented in the Field Sampling Plan Addendum (FSP) (Volume 2).

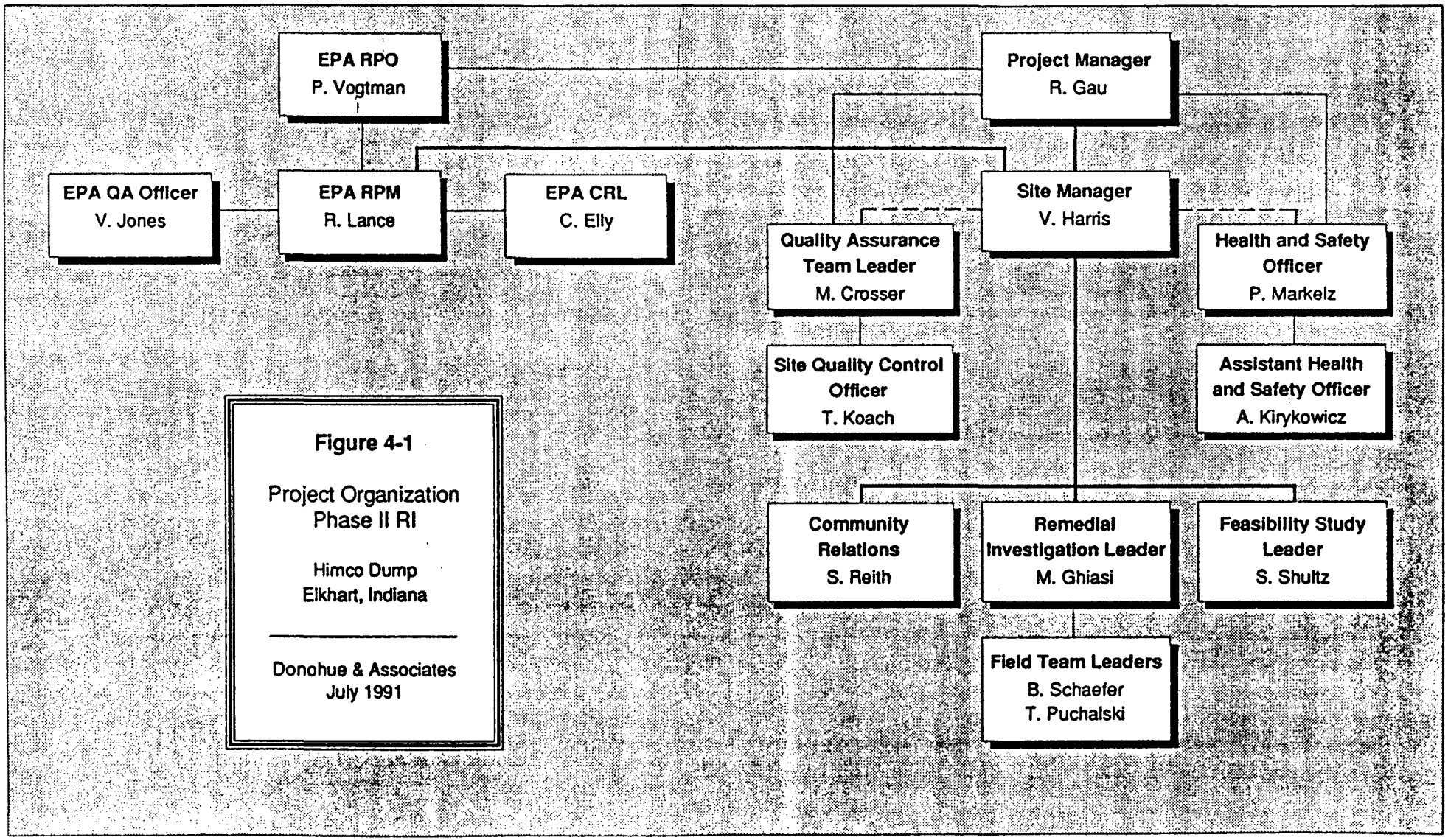
3.7 PROJECT SCHEDULE

The proposed schedule is presented in Chapter 8 of the Work Plan Addendum (Volume 1A).

The proposed schedule for the RI/FS is based on conditions discussed in the Work Plan. A six-week CLP turnaround time for RAS and SAS sample analysis with an additional three-week turnaround time for EPA data validation were assumed in developing this schedule.

4.0 PROJECT ORGANIZATION AND RESPONSIBILITY

Sections 4.0, 4.1, 4.2, 4.3, 4.4, 4.5 of the approved June 1990 Final QAPP apply. A revised project organization chart is presented in Figure 4-1. Specific duties of field team members are described in Section 3.0 of the FSP Addendum.


EPA RPO
P. Vogtman
Project Manager
R. Gau
EPA QA Officer
V. Jones
EPA RPM
R. Lance
EPA CRL
C. Elly
Site Manager
V. Harris
Quality Assurance Team Leader
M. Crosser
Health and Safety Officer
P. Markelz
Site Quality Control Officer
T. Koach
Assistant Health and Safety Officer
A. Kirykowicz
Community Relations
S. Reith
Remedial Investigation Leader
M. Ghiasi
Feasibility Study Leader
S. Shultz
Field Team Leaders
B. Schaefer
T. Puchalski
Figure 4-1
Project Organization
Phase II RI
Himco Dump
Elkhart, Indiana
Donohue & Associates
July 1991

5.6.2 Representativeness

Representativeness is a measurement of the degree to which the data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative criterion which is associated with the proper design of the sampling and analysis program. The data highly representative of this site will be achieved by performing all field sampling and measurements and laboratory analysis in a standardized manner and strictly adhering to the procedures specified in this QAPP, the Field Sampling Plan, and the Work Plan.

5.6.3 Comparability

Comparability is a qualitative criterion measuring the confidence with which one set of data can be compared with another. For this project, the data comparability will be achieved by the following:

a) Analytical results will be reported in appropriate units.

b) Same or similar sampling procedures used in the E&E 1984 field investigation and the Phase I RI will be used.

c) Same or equivalent analytical procedures used for the E&E 1984 investigation and the Phase I RI will be used.

d) Similar quality assurance and quality control requirements will be observed, since the CLP program will be used as it was in the E&E 1984 study and the Phase I RI.

5.0 QUALITY ASSURANCE OBJECTIVES

5.1 INTRODUCTION, 5.2 FIELD QA SAMPLES

Sections 5.1 and 5.2 in the approved Final QAPP, June 1990, apply with the following revision:

5.2.5 Background Samples

A background location will be included for surface water, sediment, and macroinvertebrate sample collection. Water bodies with agricultural runoff potential or industrial discharges will not be considered. A lacustrine environment similar to those water bodies located on-site will be selected by the risk assessment scientist prior to field work. Laboratory supplied "background" matrices of distilled water and sodium sulfate may need to be used if appropriate field sources cannot be located.

5.3 LABORATORY QC SAMPLES

Laboratory QC samples are specified in the applicable statement of work (SOW's); ILM01.0 (inorganics) and OLM01.1 (organics) including revisions OLM0.1.1 and more recent updates for CLP RAS procedures. Specific laboratory QC samples are included in each SAS request in Appendix A. Additional information may be found in Section 3 of the approved Final QAPjP, June 1990.

5.4 FIELD MEASUREMENT AUDITS

Section 5.4 of the approved Final QAPP, June 1990, applies.

5.5 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSES

All samples will be analyzed using the CLP. The level of QA effort for the CLP RAS analyses are specified in the CLP SOWs. In addition to routine CLP organic and inorganic analyses, SAS will be used to analyze samples for water quality parameters. These parameters and their respective QA objectives are contained in Appendix A. Soil samples will be analyzed by a SAS laboratory for physical tests by the ASTM methods contained in Appendix A.

5.6 COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY OF ANALYSES

5.6.1 Completeness

Completeness is defined as a measure of the number of samples actually collected compared to the number of samples required for characterization of an environmental condition and/or the amount of valid data obtained from the measurements system compared with the amount of data that was expected under normal conditions. This QA criterion is expressed in percentage. The completeness for sample collection will be 95 percent or better. The data completeness will be 95 percent or better for chemical analysis.

6.0 SAMPLING PROCEDURES

The media to be sampled in the RI include landfill cap soil, leachate, soil, surface water and sediment. A complete description of sampling procedures is provided in the Field Sampling Plan Addendum (Volume 2).

7.0 SAMPLE CUSTODY PROCEDURES

Sections 7.1, 7.2, 7.3, and 7.4 in the approved Final QAPP, June 1990, apply.

8.0 CALIBRATION PROCEDURES AND FREQUENCY

Sections 8.1 and 8.2 of the approved Final QAPP, June 1990, apply.

9.0 ANALYTICAL PROCEDURES

Analytical procedures to be used for the Himco Dump RI are:

- CLP RAS inorganics and organics methods for all enforcement, litigation, and evidentiary data as contained in ILM01.0 (inorganic) and OLM01.1 (organic), including revision OLM01.1.1 and more recent updates.

- SAS methods supplied by EPA Region V (contained in Appendix A) for grain size, organic carbon in soil, sulfate, chloride, nitrate and nitrite, chemical oxygen demand, total phosphorus, alkalinity, total suspended solids, total dissolved solids, total Kjeldahl nitrogen, ammonia, BOD, oil and grease, and PCB/Pesticides in fish.

- USGS procedure for bromide as contained in Appendix A - SAS Request prepared by Donohue and used for Phase I RI.

- ASTM analytical procedures for triaxial shear as contained in Appendix A - SAS Request prepared by Donohue and used for Phase I RI.

- SAS methods for total phenol and volatile suspended solid prepared by Donohue (contained in Appendix A).

9.1 ROUTINE ANALYTICAL SERVICES (RAS) LABORATORY PROCEDURES

The current EPA CLP Statement of Work (SOW) for Organics, OLM01.1, including revision OLM01.1.1 and the SOW for Inorganics Analysis, ILM01.0 (and more recent updates) specify the analytical procedures to be used. Included in the SOW are sample custody procedures, instrument calibration procedures, and frequency of calibration.

9.2 SPECIAL ANALYTICAL SERVICES (SAS) LABORATORY PROCEDURES

The analytical procedures to be used for performing the SAS analyses are specified in each SAS request in Appendix A. Specified in the SAS requests are calibration procedures, frequency of calibration, and the internal quality control checks required for each analysis.

9.3 FIELD SCREENING ANALYTICAL PROCEDURES

The procedures for field measurements are described in the SOPs contained in Appendix E. Field measurement of surface water and leachate for pH, conductivity, dissolved oxygen, and temperature will be done. Ambient air field monitoring during trenching will include volatiles by HNu meter and methane and hydrogen sulfide.

10.0 INTERNAL QUALITY CONTROL CHECKS

Sections 10.1, 10.2, 10.3, and 10.4 in the approved Final QAPP, June 1990, apply.

11.0 DATA REDUCTION, VALIDATION, AND REPORTING

Sections 11.1, 11.2, and 11.3 in the approved Final QAPP, June 1990, apply.

12.0 PERFORMANCE AND SYSTEMS AUDITS

Sections 12.1 and 12.2 in the approved Final QAPP, June 1990, apply.

13.0 PREVENTIVE MAINTENANCE

Sections 13.1 and 13.2 in the approved Final QAPP, June 1990, apply.

14.0 SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

Sections 14.1 and 14.2 in the approved Final QAPP, June 1990, apply.

15.0 CORRECTIVE ACTION

Sections 15.1, 15.2, 15.3, 15.4, 15.5 and 15.6 in the approved Final QAPP, June 1990, apply.

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

Section 16.0 in the approved Final QAPP, June 1990, applies.

APPENDIX A

SAS REQUEST FORMS

APPENDIX A

SAS REQUEST FORMS

Water Quality SAS:	Chloride, sulfate, TDS, TSS, alkalinity, TP, TKW, NH_3, NO_2 and NO_3, COD, BOD, oil and grease
Bromide SAS:	Anions, ion-exchange chromatographic, automated
Volatile Suspended Solids SAS:	Gravimetric
Total Phenol SAS:	Manual 4-AAP with distillation
Geotech SAS:	Analysis of soil samples for grain size analysis and triaxial shear
Organic Carbon SAS:	Determination of percent organic carbon in soil on air dried sample

A/P/HIMCO/AK4

WATER QUALITY SAS

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☑ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: = REGION V

B. RSCC Representative: JAN PELS

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of 21 low level groundwater samples for chloride, sulfate*, TDS, TSS, alkalinity, TP*, TKN NH3* NO2+NO3*, COD. Samples will be unfiltered, * analyses may require filtering in lab. Report results in mg/l, note if filtration required, and report as dissolved.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

21 whole AQUEOUS samples - low concentration (see Table 1 for historical data) in 4-1 liter HDPE bottles:
2L Unpreserved for Cl, SO4, TDS, TSS, alkalinity.
2L. preserved w/ H2SO4 TP, TKN, NH3, NO2+NO3, COD

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

Superfund Remedial Investigation

general WQ-1

TABLE 1

HISTORICAL LEVELS – HIMCO DUMP SAMPLES

ANALYTE	RANGE (mg/l)	POSSIBLE INTERFERENCES
Chloride	8-98	sulfate
Sulfate	5-620*	high solids
TDS	222-7830*	
TSS	unknown	
Alkalinity	150-2380*	
TP [1]	unknown	high solids
TKN [2]	unknown	
NH_3	0.02-450*	high solids – turbidity
NO_2+NO_3	<0.005-3.8	sulfide
COD	unknown	

[1] historical dissolved orthophosphate levels <0.005 – 0.60 mg/l

[2] historical dissolved organic nitrogen levels <0.005 – 750 mg/l

* well M-1

TABLE 2

HOLDING TIMES - HIMCO DUMP

ANALYTE	
BOD	48 hrs
Chloride	28 days
sulfate	28 days
TDS	7 days
TSS	7 days
alkalinity	14 days
TP	28 days
TCN	28 days
NH_3	28 days
$NO_2 + NO_3$	28 days
COD	28 days

GENERAL

WQ-4

4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment:

daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:
30 days

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): —
See specific sheets attached for each method

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): total holding times should not exceed those listed in Table 2 (from date of collection (field) to analysis). call contact if times would exceeded prior to lab receipt so recollection can occur.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.
see specific sheets attached

10. Other (use additional sheets or attach supplementary information, as needed):
See attachment 10

11. Name of sampling/shipping contact: Greg Ruechel
Phone: (414) 458-8711

GENERAL WQ-5

ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

4. Estimated date(s) of collection: _____

Estimated date(s) and method of shipment: daily by overnite carrie

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 325.1 (Colorimetric, Automated Ferricyanice, AA-I) 1983ed., or

2. EPA Method 325.2 (Colorimetric, Automated Ferricyanide, AA-II) 1983ed., or
Note: A Region V CRL Auto Analyzer Manifold is attached for Method 325.2 to correct errors in Method 325.2's manifold diagram.

3. ASTM Colorimetric Method (Manual Method) -ASTM D 512C-81, or

4. Method 407C (Potentiometric Titration) Standard Methods, 16th ed. Samples will be kept at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

For colorimetric methods (1) use a standard curve between 0 and 300 mg/l or less,(2) the calibration curve must include 5 points or more (including a zero concentration standard), and (3) samples with absorbances or peak heights greater than highest standard must be diluted and reanalyzed. For titrimetric method 1) use either 0.0141 or 0.025 N titrant, 2) automated potentiometric titrators are acceptable, 3) do not use more than 20 ml titrant for 50 ml or 100 ml sample aliquots, 4) dilute and reanalyze any sample aliquots requiring more than 20 ml titrant, 5) remove any interfering chromate, ferric iron, sulfide, and sulfite, and 6) standardize titrants daily. Obtain approval of CPMS, CRL prior to use of any other method.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The test procedure used will be clearly identified. For the colorimetric methods, bench records tabulating order of calibration standards, verification and control standards, samples, matrix spikes, titrant blanks, etc. with resulting peak height, concentration, or absorbance read-outs will be provided with copies of worksheets used to calculate results. For the titration method, any potentiometric titration curves and all bench records tabulating titrant standardization, samples, aliquot volumes, matrix spikes, etc. will be provided. Records of titrant standardization and titrant blanks will be provided. A photocopy of instrument readouts, ie. strip charts, printer tapes, etc. must be included for all analyses. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA audit results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

... Name of sampling/shipping contact: Greg Ruechel

Phone: 414 458 8711

. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
Chloride	5 mg/l	Differences in duplicate sample results are to be <5 mg/l for concentrations <50 mg/l and are to be < 10% for concentrations exceeding 50 mg/l. The significant figures to report depend on sensitivity of colorimetric curve or number of significant figures in titrant volume.
Note: These are minimum requirements Report actual detection limit used, based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
a) For Methods 325.1, 325.2, and ASTM D 512C		
Matrix Spike*	1 per group of 10 or fewer samples	85 - 115% Recovery
Lab Duplicate	" "	± (10% or 5 mg/l)
Lab Blank	" "	<5 mg/l
Calibration Verification Std.	" "	90-110% Recovery
1 Set of EPA QC Mineral Ref. Samples - 2 Concentrates	1 per sample set	85-115% Recovery
b) For Method 407C		
Same as Item IIa for Matrix Spike*, Lab Duplicate, and QC Mineral Reference Samples.		
Lab Blank (Not Titration Blank)	Beginning and end of sample set	-3 to +3 mg/l
Calibration verification Standard (Same as Titrant Standardazation)	At end of sample set	95 - 105% Recovery

*Matrix spike concentrations will be greater than 30% of the sample concentration, but spiked sample shall not exceed the working range of the standard curve or titration.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____

Estimated date(s) and method of shipment: daily by overnight Courier

6. Number of days analysis and data required after laboratory receipt of samples:

30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 375.2 (Colorimetric Methylthmol Blue) - 1983 ed.
- Note: This method requires 0.75 mg/l SO_4 in Dilution Water(See Reagent Section 6.8,

2. Method 426C of Standard Methods, 16th ed. (Turbidimetric)
- Note; this last method provides for measurement of sulfate using 2 standard curves- 1 for sulfate concentrations between 0 and 10mg/l, and 1 between 10 and 40 mg/l sulfate.

Samples will be kept at 4°C until validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Sample holding time is not to exceed 28 days from date of sample collection. Sulfate standards will be prepared daily from stock solution. Samples with absorbances or turbidities greater than that in the highest standard will be diluted and rerun. For Method 426C, 1) the reanalysis solution should contain between 20 and 40 mg/l sulfate, and 2) concentrations must be corrected for background turbidity and color per Section 5d of Method 426C using pH adjusted sample aliquots. Use only the methods specified. Calibration curves must include at least 6 points (including a zero concentration standard) for Method 375.2 and Buffer A of Method 426C.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

The test procedure used must be clearly identified. Results shall be reported as mg/l SO_4. Bench records tabulating the order of calibration standards; lab control standards, lab blanks, samples, spikes, etc., with resulting absorbances or concentration readouts, will be provided along with copies of worksheets used to calculate results. Background absorbances used for turbidity corrections must be tabulated for each sample aliquot tested. A photocopy of the instrument readout (ie. strip charts, printer tapes, etc.) must be included. All records of analysis must be legible and sufficient to calculate all concentrations and results.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

Report which samples (if any) required filtration prior to analysis.

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
Sulfate	5 mg/l	Method 375.2: Differences in duplicate sample results are to be < 5 mg/l for concentrations < 50 mg/l, and < 10% for concentrations > 50 mg/l.
		Method 426 C: Differences in duplicate sample results are to be ≤ 2 mg/l for concentrations ≤ 20 mg/l and ≤ 10% for concentrations > 20 mg/l in aliquot tested.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85-115%
Lab Duplicate	" "	± (10% or 5 mg/l) for Method 375.2
		± (10% or 2 mg/l) for Method 426C
Lab Blank (0 mg/l SO_4)	" "	< 5 mg/l - Method 375.2 -2 to +2mg/l-Buffer B of Method 426C or
Lab Blank (10 mg/l SO_4)	" "	8 to 10mg/l - Buffer A of Method 426C
Calibration Verification Standard	1 per group of 10 samples and at end of sample set	90 - 110%
1 Set of EPA QC Mineral Reference Samples	once per sample set	85-115% for each concentration.

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples shall not exceed working range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.1, 1983 ed., or

2. Method 209B, "Standard Methods", 16th ed. Samples will be kept at 4°C until sample analysis and validation of results. Holding time is 7 days from date of sample collection.

8. Specail technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1) Use standard aliquots of 100ml; however do not use sample aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller sample aliquot. 2) If the pH value is less than 4.0, raise the pH of the aliquot (using NaOH titrant) to between pH 4 and 8 and subtract the weight of sodium added from the weigth of the residue. 3) Residue will be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 the final weight is to be used for calculations. Constant weight is defined as a) less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller, or b) dried overnight (12 hours drying time) with a single weight used for calculations.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the QC reference sample lot numbers used and their true values with 95% confidence intervals. Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, duplicate samples, and reference samples will be provided with copies of work sheets used to calculate results. Dates and time of 1) determination of tare weights, 2) sample filtration, and 3) determination of residue weights and constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
TDS	20 mg/l	Difference in duplicate sample aliquots shall not exceed 2 mg for residues. Duplicate differences shall not exceed 10% for sample values greater than 200 mg/l.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. QUALITY CONTROL REQUIREMENTS Do not use any designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1. 1 set of EPA OC Mineral Reference Samples*- 2 concentration levels.	1 per sample set	85-115% Recovery
2. Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 2 mg of residue)
3. Lab Blanks (100 ml of filtered reagent water)	At least 1 per group of 10 or fewer samples	- 20 mg/l to + 20 mg/l

* Alternate reference samples must be approved by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and ~~retest~~ reanalyze samples. Contact SMO.

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.2, 1983 ed., (Gravimetric, Dried at 103° - 105° C) using glass fiber filter discs without organic binder such as: Millipore AP-40, Reeve Angel 934-AH, Gelman A/E, or equivalent. Use only membrane filter apparatus with 47 mm diameter glass fiber filter and a coarse (40-60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples will be held at 4°C until sample analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

8. Specail technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Sample aliquot volumes are selected on the basis of the following factors. a) During initial sample filtration, filtration rate should not drop rapidly, or require more than 5 minutes of filtration time. (Increase the filter area or decrease the sample volume as needed for sample reanalysis), b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200ml in volume, and c) Sample aliquots should not exceed 200ml in volume. 2. Duplicate sample aliquots will be filtered with 2 or more intervening samples. 3. Final residues are to be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 (The final weight is to be used for calculations), or dried overnight (12 hours of drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller. 4. Use only the method specified above in items 7 and 8.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify EPA QC reference sample lot numbers used and their true values and 95% confidence intervals. Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples (all in the order filtered) will be provided along with copies of worksheets used to calculate results. Dates and time of a) filtration of initial 100ml volume, b) determination of tare weights, c) sample filtration, and d) determination of constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Bucchel

Phone: 414-458-8711

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (± % or Conc.)
Suspended Solids	2-3 mg/l for 200 ml	Difference in duplicate results shall not exceed 0.5 mg for duplicate aliquots filtered.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.	sample aliquot	

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (± % or Conc.)
1) Lab Duplicates (See item 8.3 on Page 2)	1 per group of 10 or fewer samples	less than 0.5 mg for residue < less than 10% for sample. res > 5 mg
2) Lab Blanks (200 ml aliquots)	1 per group or 10 or fewer samples	-0.5 to +0.5 mg
3) 1 set of 2 EPA QC Residue Reference Samples-2 concentration levels	1 per sample set	< 5 mg/l error for concentrations < to 50 mg/l or < or = to 10% for nominal concentrations > than 50 mg/l

* Alternate reference samples must be approbed by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples.

Contact SMO

4. Estimated date(s) of collection: _____

Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days of receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) Alkalinity EPA Method 310.1 (Titrimetric, ph 4.5) 2) Standard Methods, 16th Edition, Method 403 4c and 4d.

Samples will be stored at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Samples holding time should not exceed 14 days from date of collection. Use potentiometric titration to pH 4.5 for alkalinity > 20 mg/l as $CaCO_3$. For concentrations <20 mg/l, use EPA Method 310.1 (Section 6.3) or Standard Methods, Method 403 4d. Do not use titrant volumes greater than 50ml. Obtain approval of CPMS, CRL prior to use of any other method.

Use Na_2CO_3 to standardize titrant. Standardize the pH meter and the titrant each day.

Standardize the pH meter using at least two buffers which bracket the end point.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The Test procedure used will be clearly identified. Bench records tabulating the order of analysis including pH meter calibration, titrant standardization, lab blanks, samples, lab control standards, duplicates, etc., with resulting titrant volumes or readouts will be provided along with calculation worksheets. All records will be legible and sufficient to recalculate all sample concentrations and QA audit results. Report method of titrant standardization.
EPA QC Reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (+% or Conc.)
Alkalinity	2 mg/l for low level	± 2 mg/l for Conc. < 20 mg/l $CaCO_3$
	20 mg/l for high level	± 10% for Conc. > 20 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on allowable methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits. The QA audits below will be done for each group of low-level and high-level akalinity determinations.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
lab blank	at least 1 per group of 10 or fewer samples	<10 mg/l for high-level samples tested. <2 mg/l< for low-level samples tested.
lab duplicate	at least 1 per group of 10 or fewer samples	± 10% or ± 2 mg/l
lab control sample	1 per sample set	90-110% recovery.
1 set of EPA QC mineral reference samples		

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanlyze samples.

Contact SMO

lease return this request to the Sample Management Office as soon as possible to expedite essing of your request for special analytical services. Should you have any questions need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: daily by overnight courier

Number of days analysis and data required after laboratory receipt of samples:

Laboratory should report results within 30 days after receipt of samples.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Total Phosphorus EPA Method 365.1 (Automated, Colorimetric, Ascorbic Acid)

Total Phosphorus EPA Method 365.2 (Automated, Colorimetric, Single Reagent)

Total Phosphorus EPA Method 365.4 (Block Digestor)

Samples will be preserved in the field with 1 ml/l H_2SO_4 to pH <2 and stored at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.) Check sample pH using wide-range pH paper. If the pH>2, contact CPMS, CRL for instructions:

Dilute and redigest samples with absorbances or peak heights higher than the highest standard. All standards, blanks, audits, etc. must be digested. The holding time is not to exceed 28 days from sample collection. Use only the method(s) specified above. The calibration curve must include at least 5 standards. (One of the standards must be zero concentration).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used will be clearly identified. Bench records and all records of analysis and calculations for samples, blanks, duplicates, spikes and all control checks with peak height or response and concentrations will be provided with copies of worksheets. Results will be reported as mg/l P. Any digestion log will be provided showing sample aliquots and concentrations of all samples tested. Records must be legible and sufficient to recalculate all concentrations. A photocopy of the instrument readout i.e. stripcharts, printer tapes, etc. must be included. EPA QC reference samples, or any other reference sample or initial calibration verification will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

Denote which samples (if any) required filtration prior to analysis

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Total P	0.05 mg/l	Duplicate results must agree to within 10% for concentrations > 0.5 mg/l or within 0.05 mg/l for concentrations < 0.5 mg/l
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS - Do not use designated field blanks for QA audits

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± (10% or 0.05 mg/l)
Lab Blank (Also serves as a calibration blank).	at least 1 per group of 10 or fewer samples	<0.05 mg/l
Calibration verification standard	1 per group of 10 samples and end of sample set	90% - 110%
1 set of EPA nutrient QC reference samples conc. 3&4	1 per sample set	85% - 115%

*The matrix spike concentrations will be approximately 30% or larger of sample concentrations, but spiked samples shall not exceed the working range of the standard curve.

II. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact SMO

lease return this request to the Sample Management Office as soon as possible to expedite essing of your request for special analytical services. Should you have any questions need any assistance, please call the Sample Management Office.

5/015_-0-7/87 TKN-1. Total Kjeldahl Nitrogen July 30, 1987

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples:

Laboratories shall report results within 30 days after receipt of samples

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) EPA Method 351.2 (Colorimetric, Block Digestor, AA II)

2) EPA Method 351.3 (Colorimetric, Titrimetric, or Potentiometric) (NOTE: For Method 351.3 the micro-Kjeldahl technique is not acceptable.) Samples will be preserved in the field using H_2SO_4 (1ml/L) to pH<2, samples will be stored at 4°C until analysis and validation of results.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

For all Methods: Analyze samples within 28 days after collection. Check the sample pH (wide range pH paper). If the pH>2, contact CPMS, CRL for instructions. Use nicotinic acid for the control standard. Use an organic nitrogen compound for the matrix spike. Use only the Methods specified in item 7. Method 351.3 requires distillation separation, prior to all final ammonia measurements.
For Method 351.3: Use only the Colorimetric method for samples containing less than 1 mg N/l.
For Colorimetric Methods (351.2 and 351.3): Use at least five calibration standards (including a zero concentration standard). Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Potentiometric Method (351.3): Use at least four calibration standards. Dilute and reanalyze samples with concentrations that exceed the highest calibration standard.
For the Titrimetric Method (351.3): Standardize the titrant each day. Include records of indicator blank.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify the test procedure and options used. Provide bench records and all records of calibration, analyses, and calculations for standards, samples blanks, any titration indicator blanks, duplicates, spikes, controls, etc. Include absorbances, peak heights, responses, concentrations, etc. for each measurement. Include digestion logs showing sample volumes and dilutions for all samples. Identify organic nitrogen compound used for matrix spikes. Records must be legible and sufficient to recalculate all concentrations and QA audit results. Provide photocopies of all instrument readouts (i.e. stripcharts, print-outs, etc). Report results as mg N/l. Identify the compound used for the matrix spike.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "tru" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

TKN-1

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (+% or Conc.)
TKN	0.1 mg N/l	Duplicate sample results must agree within 0.1 mg/l for concentrations <1 mg/l and within 10% for concentrations > or = to 1 mg/l
NOTE: These are minimum requirements. Report the actual detection limit used based on allowable methodology options.		

II. QC REQUIREMENTS Do not use designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Control standards (Nicotinic Acid)	one per set	70 - 110% recovery
Matrix spike*	one per group of 10 or fewer samples	85 - 115% recovery
Lab duplicate	" "	± (10% or 0.1 mg N/l)
Lab blank	" "	± 0.1 mg N/l
Calibration verification Standard	" " and at the end of the set	90 - 110%
1 set of EPA QC nutrient reference samples conc. 3 and 4.	one per set	85 - 115%

*Matrix spike concentration will be greater than 30% of the sample concentration but will not exceed the highest calibration standard. Matrix spikes will be prepared from an organic nitrogen compound.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples.

Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1) EPA Method 350.1 (Automated Phenate), or

2) EPA Method 350.3 (Potentiometric, Ion Selective Electrode).

Samples will be stored at 4° C until analysis and validation of results. Sample aliquots will be preserved in the field with sulfuric acid (1 ml/l to pH < 2).

The working concentration range of Method 350.1 Auto Analyzer should be 0.1 to 10 mg/l NH_3-N or lesser concentration.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Check sample pH (wide range pH paper). If pH > contact Jay Thakkar, CPMS, CRL for instructions. Dilute and rerun samples with peak heights or concentrations higher than the highest standard. The holding time is not to exceed 28 days from sample collection. All solutions should be made with amonia-free water. For Method 350.3 calibrate the electrometer with standards in order of increasing concentration of ammonia. The pH of the solution after the addition of NAOH must be above 11. Use only the method(s) specified above. Standard curve for Method 350.1 must include at least 5 standards (one of which is zero concentration). Standard curve for Method 350.3 must include at least 4 standards between 0.1 and 10.0 mg/l NH3-N. All standards, blanks, dilution water, and diluted samples must be acidified with 1 ml/l H_2SO_4.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:

The test procedure used will be clearly identified. Bench records tabulating the order of calibration standards, lab blanks, samples, lab control standards, spikes, duplicate, etc. with resulting peak heights, millivolts, or concentration readouts, will be provided along with copies of worksheets used to calculate ammonia results. If Method 350.3 is used, the standard curve should be provided. A photocopy of the instrument readout i.e. strip charts, printer tapes, etc. must be included. All records analyses and calculation must be legible and sufficient to recalculate all concentrations. Results are to be in mg/-N per liter.
EPA QC reference samples, or any other reference sample or initial calibration verification. will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):
note if filtration needed, report which samples were filtered

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

5/013G-0-6/87 NH3-2 -3- Analysis of ammonia
June 30, 1987

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
Ammonia	0.1 mg/l-N	Duplicate results must agree to within 10% for concentrations > 1mg/l or to within 0.1mg/l for concentrations <1 mg/l Results will be reported to the nearest 0.05 mg/l and to 2 significant figures for concentrations exceeding 1/mg/l-N.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

GENERAL STATEMENT

II. QC REQUIREMENTS - Do not use designated field blanks for QA Audits.

a) For Method 350.1

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	at least 1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	at least 1 per group of 10 or fewer samples	± 10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤0.1 mg/l
Calibration verification	1 per group of 10 samples	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2	1 per sample set	85% - 115%

b) For Method 350.3

Lab Duplicate	at least 1 per group of 10 or fewer samples	10% or 0.1 mg/l
Lab Blank	at least 1 per group of 10 or fewer samples	≤ 0.1 mg/l
Calibration verification standard	1 per 10 samples and end of set	90% - 110%
1 set of EPA QC Nutrient reference samples. Conc. 1 & 2.	1 per sample set	85% - 115%

*Matrix spike concentrations will be greater than 30% of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

4. ~~Estimated~~ date(s) of collection: _____

NO_2-NO_3-1 5. ~~Estimated~~ date(s) and method of shipment: daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
1) EPA Method 353.1 (colorimetric, automated hydrazine reduction).
2) EPA Method 353.2 (colorimetric, automated cadmium reduction).
3) EPA Method 353.3 (colorimetric, manual cadmium reduction).

For all methods:
Samples will be stored at 4°C until analysis and validation of results. Samples will be preserved in the field with sulfuric acid (1 ml/l) to pH<2. The analytical working range shall not exceed 0.1 to 10.0 mg/l N.

For Methods 353.2 or 353.3: If more than one reduction column is used separate calibrations, QA audits, and records are required for each column. The column used must be identified for each analytical result.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
Analyze the samples within 28 days after collection. Check the sample pH (wide range pH paper is acceptable). If the pH>2 contact CPMS, CRL for instructions. Use only the methods specified in item 7. Obtain approval of CPMS, CRL before using any other method.
For Methods 353.2 and 353.3: After checking the pH it is recommended that the laboratory check for residual chlorine (or oxidizing reagents) and sulfide using test strips such as starch iodide and lead acetate papers. Contact CPMS, CRL if these interferences are present; however, the laboratory must remove these interferences prior to analysis. The laboratory must also minimize interferences due to metals in order to prolong column life. (See Section 7.1.2 of method 353.3) It is suggested that the laboratory may dilute samples up to ten-fold prior to analysis (Section 7.4 of Method 353.3) provided that the final analytical working range does not exceed 0.1 to 10.0 mg/l N.
For all methods: Neutralize samples to pH 5-9 (or to phenolphthalein color end-point) prior to analysis. Dilute and reanalize the neutralized samples if the concentrations exceed that of the highest standard. Use at least five calibration standards (including a zero standard). Prepare the lab blank using 1 ml of H_2SO_4/l. Neutralize and analyze it like a sample.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion:
The test procedure used must be clearly identified. Bench records tabulating the order of calibration standards, lab control standards, lab blanks, samples, spikes, duplicates, etc., with resulting absorbances or concentration readouts will be provided. Worksheets used to calculate results will be included. Any sample treatment to remove interferences will be documented. The laboratory shall submit photocopies of the instrument readout (strip-charts, printer tapes, etc.) All records of analysis and calculations must be legible and sufficient to recalculate all concentrations. Results are to be reported as mg N/l.
EPA QC reference samples, or any other reference sample or initial calibration verification will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):
report which samples (if any) required preliminary

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414 458 8711

NO_2+NO_3-1

5/0146-0-6/87 NO_2+NO_3 Analysis of Nitrate 6/29/87

I. DATA REQUIREMENTS NO_2+NO_3-2

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
Nitrate + Nitrite	0.10 mg/l as N	Duplicate results must be within 10% for concentrations >1mg/l or within 0.1 mg/l for concentrations ≤ 1mg/l Results will be reported to the nearest 0.1 mg/l for conc. less than 1.0 mg/l and to 2 significant figures for conc. exceeding 1 mg/l-N.
Note: These are minimum requirements. Report actual detection limits used based on allowable methodology options.		

II. QC REQUIREMENTS - Do not use any designated field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix Spike*	1 per group of 10 or fewer samples	85% - 115%
Lab Duplicate	1 per group of 10 or fewer samples	±(10% - or 0.1 0 mg/l)
Lab Blank (1ml/l H_2SO_4)	2 per sample set	<0.1 mg/l
Calibration verification standard	1 per group of 10 or fewer samples and at end of run	90% - 110%
Calibration blank	1 per group of 10 samples or less	< 0.1 mg/l
1 set of EPA Nutrient QC reference samples-conc. 1 and 2, or EPA F/NO_3 QC sample, WS series Conc. 1 and 2	1 per sample set	85% - 115%

*Matrix spike concentrations will be 30% or larger, of sample concentrations, but spiked samples should not exceed working concentration range of standard curve.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

NO_2+NO_3-2

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: daily by overnight courier

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 410.1 (Titrimetric, Mid-level) for COD ≥ 50 mg/l.
EPA Method 410.2 (Titrimetric, Low-level) for COD < 50 mg/l.
Use Section 7.1 of Method 410.3 if chloride concentration exceeds 2000 mg/l in a sample. If titration blank is necessary for each different amount of mercuric sulfate used for inhibition of chloride interference, SAS Packing Lists will note the samples requiring assessment of chloride interferences. Measurement of chloride will be done using any method of "Standard Methods",16th ed., or "EPA Methods for Chemical Analysis of Water and Wastes", 1983 ed., whenever possible chloride interference is noted.
Samples will be preserved with 1 ml of H_2SO_4 to pH less than 2 and kept at 4°C until sample analysis and validation of results are completed. Holding time is not to exceed 2 days from date of sample collection.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Check sample pH (wide range pH paper). If pH>2, contact CPMS, CRL for further instructions.
2. Use a) 50 ml sample aliquots for both methods, b) 0.250 N $K_2Cr_2O_7$ reagent and 0.25 N ferrous ammonium sulfate titrant for Method 410.1, and c) 0.0250 N $K_2Cr_2O_7$ reagent and 0.025 N ferrous ammonium sulfate titrant for Method 410.2.
3. Dilute and reanalyze (by Method 410.1) any samples with COD values > 800 mg/l or titrant volumes < 5.0 ml. Reanalyze samples (by Method 410.1) if initial sample values are > 50 mg/l COD by Method 410.2. Reanalyze samples (by Method 410.2) if initial sample values are < 50 mg/l COD by Method 410.1.
4. Any sample aliquots < 50 mls will be diluted to 50 mls so that the COD reaction mixture will be 50% H_2SO_4/ 50% water by volume.
5. Titration blanks will be determined, at least in duplicate each day of analysis and will not differ more than ± 0.1 ml titrant for Method 410.1 and ± 1.0 ml titrant for Method 410.2.
6. Separate sets of QA Audits will be performed for each method, if both methods are used.
7. Use potassium hydrogen phthalate as a matrix spike compound. Use 20 mg/l matrix spike concentration for Method 410.2.
8. Samples will be refluxed for at least 2 hours.
9. Homogenize sample aliquots, as necessary, to obtain sample aliquots of representative suspended solids.
10. Use only the method specified.

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (± % or Conc.)
COD (Method 410.1)	50 mg/l	Method 410.1: Differences in sample duplicates are to be < or = to 0.2 ml titrant or ≤ 8 mg/l for concentrations ≤ 80 mg/l and ≤ 10% for COD concentrations exceeding 80 mg/l.
COD (Method 410.2)	5 mg/l	Method 410.2: Differences in sample duplicate results are t be ≤ 1.0 ml titrant or ≤ 4 mg/ for concentrations less than 40 mg/l and are to be ≤ 5 mg/l for concentrations between 40 50 mg/l.
NOTE: These are minimum requirements. Report actual detection limits used based on specified methodologies.		

II. QC REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Matrix spike (KHP) Method 410.1* Method 410.2(Use 20 mg/l spike)	at least 1 per group of 10 or fewer samples	85 - 115% Recovery (410.1) 75 - 125% Recovery (410.2)
Lab duplicate		Diff ≤ (8 mg/l or 10%) (410.1) Diff ≤ (4 mg/l - 5 mg/l) (410.2)
Titration blank (used for calculation of results)	at least 2 per sample set for each method used	Diff in titrant volumes shall not exceed 0.1 ml for 410.1 and 1.0 ml for 410.2
1 set of EPA QC Demand Reference samples - 2 concentration levels	1 per sample set for each method used	90 - 110% Recovery or ≤ 8 mg/l error for 410.1 and ≤ 5 mg/l error for 410.2 in aliquot - tested

* - Matrix spike will be greater than 30% of the sample concentration, but spiked sample shall not exceed 800 mg/l for Method 410.1.

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples. Contact SMO

ase return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

TOC-3

12. Data Requirements

Parameter	Detection Limit	Precision Desired (±% or Concentration)
% organic carbon	0.10 %, report actual achieved if smaller	± 20 % on lab duplicates

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
prep blank	1 in 10, max of 2	≤ 0.1 %
duplicate	1 in 5, max of 4	≤ 20 % rpd or ≤ 0.2 % at concentrations of 0.1 % - 0.3 %
positive control (lab determines)	1 in 10, max of 2	85 - 115 %
instrument calibration checks + blanks	1 in 10	90 - 110 % recovery for Cal. check ≤ 0.1 % total carbon for assumed routine sample weig

14. Action Required if Limits are Exceeded

Take corrective action, reanalyze samples, contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

ATTACHMENT I

. Determination of organic carbon(%) in soil, using sub-aliquots of air-dried soil, passed through a 100 mesh to 140 mesh screen. All of the sub-aliquot must pass the screen. Applicable organic carbon concentration range of interest is 0.1% to 2% (or larger) in soil, (dry weight basis). Laboratory may report lower concentration values.

Test procedures used for determining soil shall be the 1) dry combustion (resistence furnace), 2) Dry combustion (induction furnace), 3) Dry combusiton (automated methods), or 4) Wet combustion (combustion train) methods of analysis specified by Table 29-1 of "Methods of Soil Analyses," Part 2 - Chemical and Microbiological properties, 2nd ed., 1982, American Society of Agronomy, and Soil Science Society of America, Madison, Wisconsin. Copies of this copyrighted material are not being provided, because no laboratory doing organic carbon analysis of soil should be without it.

Any automated dry combustion test procedure used must provide results consistent with the other 3 methodologies and must be consistent with the requirements of Chapter 29, Sections 29-1, 29-2, and 29-3, "Methods of Soil Analysis" (MSA) Part II, 2nd ed., as appropriate. Soils can be calcerous or noncalcerous soils, with varying amounts of organic carbon. Soils determined may be subsurface as well as surface soils. If peat or muck soils are ever encountered, the laboratory will provide with the case narrative, limitations of any sample results and any solutions to problems encountered. This is also true for any other problem sample types encountered.

The laboratory, providing organic carbon analysis data, will provide information with the case narrative concerning methodology, instrumentation, and specific QA practices used for the set of soils tested. Requested information is detailed in items #8, and #9 of this SAS.

TOC-5

ATTACHMENT 7
Analytical Methods - Organic Carbon in Soil

7a. Sample Preparation: Representative sub-aliquot of air-dried soil (see % solids SAS) screened through 100 or 140 mesh as appropriate. All of the sub-aliquot must pass this screen.

b. Test for Presence of Inorganic Carbon, MSA, Part II, Section 29-3.3.1. Place finely ground soil on a spot plate, and moisten with a few drops of water. Add 4 N HCl dropwise to the wetted sample and observe any effervescence. Allow sufficient time for dolomite to react (~5 min). If inorganic carbon is absent proceed with Total Carbon in items #7c, or 7d below. If inorganic carbon is present, or the test is not definitive, proceed with tiems #7e, or #7f prior to Total Carbon measurements of Item #7c or #7d.

c. Total Carbon (Dry Combustion), MSA, Part II, Section 29-2.2.2. Use this as a guide for instrumental specifications. Instrument must test solid sample directly. Illustrative examples of this methodology are:

1) Total Carbon (Dry Combustion - Medium Temperature Resistance Furnace), MSA, Part II, Section 29-2.2.3.

2) Total Carbon (Dry Combusiton - High Temperature Induction Furnace), MSA, Part II, Section 29-2.2.4.

3) Total Carbon (Dry Combusiton - Other Instrumental Methods), MSA, Part II, Section 29-2.2.5. Any other instrumentation such as this must be justified and provide results as precise and accurate as the results from Sections 29-2.2.3, and 29-2.2.4.

d. Total Carbon (Wet Digestion), MSA, part II, Section 29-2.3.2 Soil digested in 60:40 mixture of sulfuric acid and phosphoric acid (containing $K_2Cr_2O_7$). CO_2 evolved is absorbed and weighed, or absorbed in standard base and titrated.

1) Specific examples are found in MSA, Part II, Figure 29-2, Figure 29-3, and Section 29-2.3.3.

e. Pretreatment prior to Dry Combustion, MSA, Part II, Section 29-3.3.3. Inorganic carbon is removed by treating sample in a combustion boat, with 5% sulfurous acid (H_2SO_3). After several hours, remove the excess H_2SO_3 by leaving overnight in an evacuated dessicator. Read citation for further details.

f. Pretreatment prior to Wet Digestion, MSA, Part II, Seciton 29-3.3.2. Inorganic carbon is removed by sulfuric acid - ferrous sulfate reagent in apparatus used for total carbon (Wet Digestion) prior to Total Carbon measurement. See citation for further details.

- (cont. next page) -

ATTACHMENT 7 (Cont.)

g. Use only the methods specified above or obtain approval of CPMS, CRL prior to use of other method. Test procedure description, and description of specific measurement principles including equivalency to each of the 10 items of Figure 29-1 of MSA, part II and sample pretreatmenst of Section 29-3, MSA, Part II.

h. Laboratory performing Total Carbon determinations must use and have a recognized procedure for removal of any inorganic carbon in sample.

ATTACHMENT 8

A variety of apparatus, instrumentation, sample preparation systems and read-outs can be used. It is the responsibility of the laboratory to provide appropriate QC audits and QC data with each set of samples tested.

If instrumentation requires calibration, provide calibration curve, including zero concentration standard and preparation blanks. Provide positive control (a test sample prepared independently from calibration standards) that provides a measure of accuracy of system. This should be done for all systems including grarmetric read-outs.

ATTACHMENT 9
Analytical Results Required

As part of Case Narrative, attach description of test procedure and instrumentation used for measurement of Total C and removal of any Inorganic C. Test procdure description must include sufficient information that the nature of specific analytical result deliverables can be determined including QC audits. In Case Narrative, discuss any problem type samples (including peat or muck soils), limitations on any sample results, and soultion taken to resolve any problems. A sample preparation log will be provided, as appropriate.

Bench record tabulating any order of any sample weights and tare weights of absorbed CO_2, instrument calibrations, blanks, QA audits, etc., must be provided along with copies of any worksheets used to calculate results. Include copies of any instrument readouts. All must be legible. Report results as % organic Carbon on a dry weight basis (103-105°).

ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

 Geotech-27

Specific gravity, $T_x/20\,C = K \times$ specific gravity, T_x/T_x

where:

K = a number found by dividing the relative density of water at temperature T_x by the relative density of water at 20°C. Values for a range of temperatures are given in Table 1.

9.3 When it is desired to report the specific gravity value based on water at 4°C, such a specific gravity value may be calculated by multiplying the specific gravity value at temperature T_x by the relative density of water at temperature T_x.

9.4 When any portion of the original sample of soil is eliminated in the preparation of the test sample, the portion on which the test has been made shall be reported.

10. Precision and Bias

10.1 Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the No. 4 (4.75-mm) sieve are given as follows (Note 8):

Material and Type Index	Standard Deviation[A]	Acceptable Range of Two Results (percent of mean)[A]
Single-operator precision:		
Cohesive soils	0.021	0.06
Noncohesive soils	[B]	[B]
Multilaboratory precision:		
Cohesive soils	0.056	0.16
Noncohesive soils	[B]	[B]

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

[B] Criteria for assigning standard deviation values for non-cohesive soils are not available at the present time.

NOTE 8—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

TOC-soil-1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: = V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Determination of organic carbon (%) in soil on air dried sample screened through a 100 or 140 mesh sieve. Applicable concentrations 0.1 - 2.0 %, see Attachment 1

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

23 low level soil samples

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

Superfund remedial investigation

TOC-2

4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment: one shipment at end of project by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): —
See Attachment 7

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
See Attachment 8

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.
See Attachment 9

10. Other (use additional sheets or attach supplementary information, as needed):
See attachment 10

11. Name of sampling/shipping contact: Greg Ruecke'
Phone: (414) 458-8711

Geotech -23

where:

D = diameter of particle, mm,

n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)),

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of soil particles, and

G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}\,[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm³, and

A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm³

A = 27.8 cm²

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

17. Graph

17.1 When the hydrometer analysis is performed, a graph

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TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01278	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01256	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles.

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular.

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(3) Silt size, 0.074 to 0.005 mm ..... %
(4) Clay size, smaller than 0.005 mm ..... %
  Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-μm)	.....
No. 200 (75-μm)	.....

HYDROMETER ANALYSIS

0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

Designation: D 854 - 83

American Association State Highway and Transportation Officials Standard AASHTO No.: T 100

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Standard Test Method for Specific Gravity of Soils[1]

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75-mm) sieve, the method outlined in Test Method C 127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values (Note 1). When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D 422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00-mm) sieve.

NOTE 1—The weighted average specific gravity should be calculated using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}}$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the No. 4 (4.75-mm) sieve,
R_1 = percent of soil particles retained on the No. 4 sieve,
P_1 = percent of soil particles passing the No. 4 sieve,
G_1 = apparent specific gravity of soil particles retained on the No. 4 sieve as determined by Test Method C 127, and
G_2 = specific gravity of soil particles passing the No. 4 sieve as determined by this test method.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]
C 670 Practice for Preparing Precision Statements for Test Methods for Construction Materials[2]
D 422 Method for Particle-Size Analysis of Soils[3]
E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]

3. Definition

3.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature (per Definitions E 12).

4. Significance and Use

4.1 The specific gravity of a soil is used in almost every equation expressing the phase relationship of air, water, and solids in a given volume of material.

4.2 The term "solid particles," as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not very soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, etc.), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity of less than one, typically require special treatment or a qualified definition of specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL (Note 2). The stopper shall be of the same material as the bottle, and of such size and shape that it can be easily inserted to a fixed depth in the neck of the bottle, and shall have a small hole through its center to permit the emission of air and surplus water.

NOTE 2—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference, but in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 28, 1983. Published January 1984. Originally issued as D 854 - 45. Last previous edition D 854 - 58 (1979).

[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vol 04.08.
[4] *Annual Book of ASTM Standards*, Vol 15.05.

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5.2 *Balance*—Either a balance sensitive to 0.01 g for use with the volumetric flask, or a balance sensitive to 0.001 g for use with the stoppered bottle.

6. Calibration of Pycnometer

6.1 The pycnometer shall be cleaned, dried, weighed, and the weight recorded. The pycnometer shall be filled with distilled water (Note 3) essentially at room temperature. The weight of the pycnometer and water, W'_a, shall be determined and recorded. A thermometer shall be inserted in the water and its temperature T, determined to the nearest whole degree.

NOTE 3—Kerosine is a better wetting agent than water for most soils and may be used in place of distilled water for oven-dried samples.

6.2 From the weight W'_a determined at the observed temperature T, a table of values of weights W'_a shall be prepared for a series of temperatures that are likely to prevail when weights W'_b are determined later (Note 4). These values of W'_a shall be calculated as follows:

$$W'_a \text{ (at } T_x) = (\text{density of water at } T_x/\text{density of water at } T) \times (W'_a \text{ (at } T) - W'_t) + W'_t$$

where:

W'_a = weight of pycnometer and water, g,
W'_t = weight of pycnometer, g,
T = observed temperature of water, °C, and
T_x = any other desired temperature, °C.

NOTE 4—This method provides a procedure that is most convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when weights W'_a and W'_b are taken, requires considerable time. It is much more convenient to prepare a table of weights W'_a for various temperatures likely to prevail when weights W'_b are taken. It is important that weights W'_a and W'_b be based on water at the same temperature. Values for the relative density of water at temperatures from 18 to 30°C are given in Table 1.

7. Sampling

7.1 The soil to be used in specific gravity test may contain its natural moisture or be oven-dried. The weight of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

7.2 *Samples Containing Natural Moisture*—When the sample contains its natural moisture, the weight of the soil, W_s, on an oven-dry basis shall be determined at the end of the test by evaporating the water in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5). Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422 (Note 6).

7.3 *Oven-Dried Samples*—When an oven-dried sample is to be used, the sample shall be dried for at least 12 h, or to constant weight, in an oven maintained at 230 ±9°F (110 ± 5°C) (Note 5), cooled in a desiccator, and weighed upon removal from the desiccator. The sample shall then be soaked in distilled water for at least 12 h.

NOTE 5—Drying of certain soils at 110°C may bring about loss of moisture of composition or hydration, and in such cases drying shall be done, if desired, in reduced air pressure and at a lower temperature.

NOTE 6—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-mL flask is needed as the pycnometer.

TABLE 1 Relative Density of Water and Conversion Factor K For Various Temperatures

Temperature, °C	Relative Density of Water	Correction Factor K
18	0.9986244	1.0004
19	0.9984347	1.0002
20	0.9982343	1.0000
21	0.9980233	0.9998
22	0.9978019	0.9996
23	0.9975702	0.9993
24	0.9973286	0.9991
25	0.9970770	0.9989
26	0.9968156	0.9986
27	0.9965451	0.9983
28	0.9962652	0.9980
29	0.9959761	0.9977
30	0.9956780	0.9974

8. Procedure

8.1 Place the sample in the pycnometer, taking care not to lose any of the soil in case the weight of the sample has been determined. Add distilled water to fill the volumetric flask about three-fourths full or the stoppered bottle about half full.

8.2 Remove entrapped air by either of the following methods: (1) subject the contents to a partial vacuum (air pressure not exceeding 100 mm Hg) or (2) boil gently for at least 10 min while occasionally rolling the pycnometer to assist in the removal of the air. Subject the contents to reduced air pressure either by connecting the pycnometer directly to an aspirator or vacuum pump, or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask. Cool samples that are heated to room temperature.

8.3 Fill the pycnometer with distilled water, clean the outside and dry with a clean, dry cloth. Determine the weight of the pycnometer and contents, W'_b, and the temperature in degrees Celsius, T_x, of the contents as described in Section 6.

9. Calculation and Report

9.1 Calculate the specific gravity of the soil, based on water at a temperature T_x, as follows:

$$\text{Specific gravity, } T_x/T_x = W'_s/[W'_a + (W'_s - W'_b)]$$

where:

W'_s = weight of sample of oven-dry soil, g,
W'_a = weight of pycnometer filled with water at temperature T_x (Note 7), g,
W'_b = weight of pycnometer filled with water and soil at temperature T_x, g, and
T_x = temperature of the contents of the pycnometer when weight W'_b was determined, °C.

NOTE 7—This value shall be taken from the table of values of W'_a prepared in accordance with 6.2, for the temperature prevailing when weight W'_b was taken.

9.2 Unless otherwise required, specific gravity values reported shall be based on water at 20°C. The value based on water at 20°C shall be calculated from the value based on water at the observed temperature T_x, as follows:

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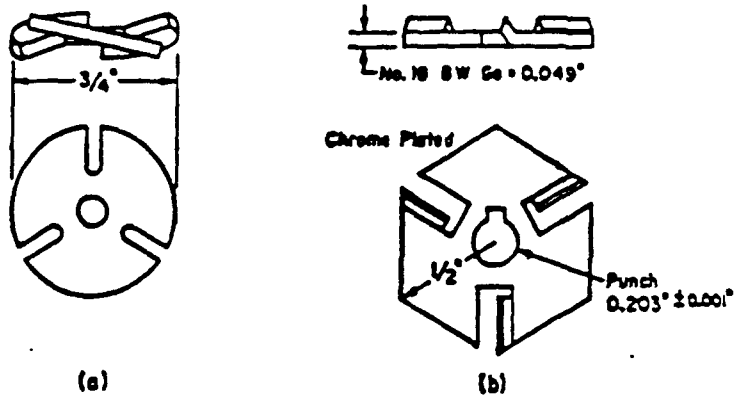


Metric Equivalents					
in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles

3-in. (75-mm)
2-in. (50-mm)
1½-in. (37.5-mm)
1-in. (25.0-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 20 (850-μm)
No. 40 (425-μm)
No. 60 (250-μm)
No. 140 (106-μm)
No. 200 (75-μm)

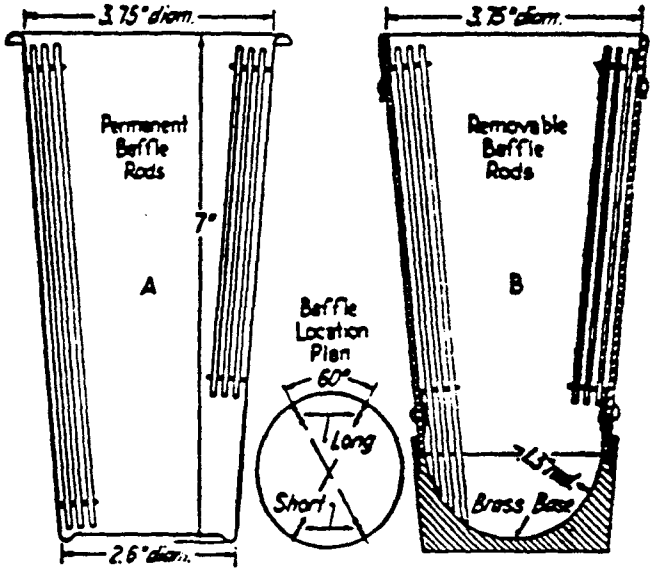
NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)
1½-in. (37.5-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 8 (2.36-mm)
No. 16 (1.18-mm)
No. 30 (600-μm)
No. 50 (300-μm)
No. 100 (150-μm)
No. 200 (75-μm)

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.



Metric Equivalents			
in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

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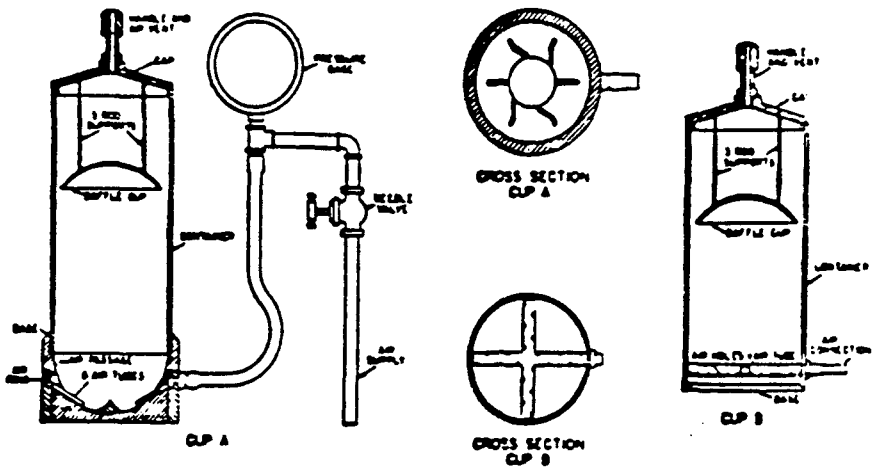

FIG. 3 Air-Jet Dispersion Cups of Apparatus B

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Larger Particles in (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

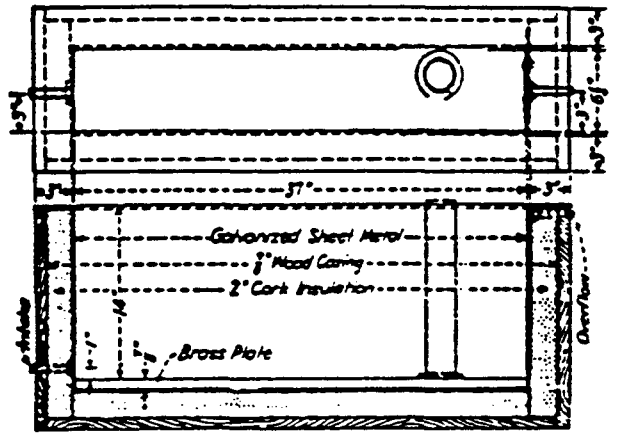

FIG. 4 Insulated Water Bath

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

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HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil - water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns.

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Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

TABLE 1 Values of Correction Factor, a, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

- a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),
- P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,
- R = hydrometer reading with composite correction applied (Section 7),
- W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,
- G = specific gravity of the soil particles, and
- G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for a is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)]} \times L/T$$

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content. After batching, store the material in a covered container for at least 16 h prior to compaction. Specimens may be molded to the desired density by either: (1) kneading or tamping each layer until the accumulative weight of the soil placed in the mold is compacted to a known volume or (2) by adjusting the number of layers, the number of tamps per layer, and the force per tamp. Scarify the top of each layer prior to the addition of material for the next layer. The tamper used to compact the material shall have an area in contact with the soil equal to or less than ½ the area of the mold. After a specimen is formed, with the ends perpendicular to the longitudinal axis, remove the mold and determine the mass and dimensions of the specimen using the devices described in 5.9 and 5.11. Perform one or more water content determinations on excess material used to prepare the specimen in accordance with Method D 2216.

NOTE 9—It is common for the unit weight of the specimen after removal from the mold to be less than the value based on the volume of the mold. This occurs as a result of the specimen swelling after removal of the lateral confinement due to the mold.

NOTE 10—Experience indicates that it is difficult to compact, handle, and obtain valid results with specimens that have a degree of saturation that is greater than about 90 %.

7. Procedure

7.1 Place the membrane on the membrane expander or, if it is rolled onto the specimen, roll the membrane onto the cap or base. Place the specimen on the base. Place the rubber membrane around the specimen and seal it at the cap and base with O-rings or other positive seals at each end. A thin coating of silicon grease on the vertical surfaces of the cap or base will aid in sealing the membrane.

7.2 With the specimen encased in the rubber membrane, which is sealed to the specimen cap and base and positioned in the chamber, assemble the triaxial chamber. Bring the axial load piston into contact with the specimen cap several times to permit proper seating and alignment of the piston with the cap. When the piston is brought into contact the final time, record the reading on the deformation indicator. During this procedure, take care not to apply an axial stress to the specimen exceeding approximately 0.5 % of the estimated compressive strength. If the weight of the piston is sufficient to apply an axial stress exceeding approximately 0.5 % of the estimated compressive strength, lock the piston in place above the specimen cap after checking the seating and alignment and keep locked until application of the chamber pressure.

7.3 Place the chamber in position in the axial loading device. Be careful to align the axial loading device, the axial load-measuring device, and the triaxial chamber to prevent the application of a lateral force to the piston during testing. Attach the pressure-maintaining and measurement device and fill the chamber with the confining liquid. Adjust the pressure-maintaining and measurement device to the desired chamber pressure and apply the pressure to the chamber fluid. Wait approximately 10 min after the application of chamber pressure before continuing the test.

NOTE 11—In some cases the chamber will be filled and the chamber pressure applied before placement in the axial loading device.

NOTE 12—Make sure the piston is locked or held in place by the axial loading device before applying the chamber pressure.

NOTE 13—The purpose of the waiting period is to allow the specimen to stabilize under the chamber pressure prior to application of the axial load.

7.4 If the axial load-measuring device is located outside of the triaxial chamber, the chamber pressure will produce an upward force on the piston that will react against the axial loading device. In this case, start the test with the piston slightly above the specimen cap, and before the piston comes in contact with the specimen cap, either: (1) measure and record the initial piston friction and upward thrust of the piston produced by the chamber pressure and later correct the measured axial load, or (2) adjust the axial load-measuring device to compensate for the friction and thrust. If the axial load-measuring device is located inside the chamber, it will not be necessary to correct or compensate for the uplift force acting on the axial loading device or for piston friction. In both cases record the initial reading on the deformation indicator when the piston contacts the specimen cap.

7.5 Apply the axial load to produce axial strain at a rate of approximately 1 %/min for plastic materials and 0.3 %/min for brittle materials that achieve maximum deviator stress at approximately 3 to 6 % strain. At these rates, the elapsed time to reach maximum deviator stress will be approximately 15 to 20 min. Continue

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the loading to 15 % axial strain, except loading may be stopped when the deviator stress has peaked then dropped 20 % or the axial strain has reached 5 % beyond the strain at which the peak in deviator stress occurred.

7.6 Record load and deformation values at about 0.1, 0.2, 0.3, 0.4, and 0.5 % strain; then at increments of about 0.5 % strain to 3 %; and, thereafter at every 1 %. Take sufficient readings to define the stress - strain curve; hence, more frequent readings may be required in the early stages of the test and as failure is approached.

NOTE 14—Alternate intervals for the readings may be used provided sufficient points are obtained to define the stress - strain curve.

7.7 After completion of the tests, remove the test specimen from the chamber. Determine the water content of the test specimen in accordance with Method D 2216 using the entire specimen, unless representative cuttings are obtained for this purpose, as in the case of undisturbed specimens. Indicate on the test report whether the water content sample was obtained before or after the shear test, as required in 9.1.2.

7.8 Make a sketch, or take a photo, of the test specimen at failure and show the slope angle of the failure surface if the angle is visible and measurable.

8. Calculations

8.1 Calculate the axial strain, ϵ (expressed as a decimal), for a given applied axial load, as follows:

$$\epsilon = \Delta L / L_o$$

where:

ΔL = change in length of specimen as read from deformation indicator, and

L_o = initial length of test specimen minus any change in length prior to loading.

8.2 Calculate the average cross-sectional area, A, for a given applied axial load as follows:

$$A = A_o / (1 - \epsilon)$$

where:

A_o = initial average cross-sectional area of the specimen, and

ϵ = axial strain for the given axial load (expressed as a decimal).

NOTE 15—In the event that the application of the chamber pressure results in a change in the specimen length, A_o should be corrected to reflect this change in volume. Frequently, this is done by assuming that lateral strains are equal to vertical strains. The diameter after volume change would be given by $D = D_o(1 - \Delta L/L)$.

8.3 Calculate the principal stress difference (deviator stress), $\sigma_1 - \sigma_3$, for a given applied axial load as follows:

$$\sigma_1 - \sigma_3 = P/A$$

where:

P = measured applied axial load (corrected for uplift and piston friction, if required see 7.4), and

A = corresponding average cross-sectional area.

8.4 *Stress - Strain Curve*—Prepare a graph showing the relationship between principal stress difference (deviator stress) and axial strain, plotting deviator stress as ordinate and axial strain (in percent) as abscissa. Select the compressive strength and axial strain at failure in accordance with the definitions in 3.2.1 and 3.2.2.

8.5 *Correction of Strength Due to Stiffness of Rubber Membrane*—Assuming units are consistent, the following equation, or other acceptable equations, shall be used to correct the principal stress difference or deviator stress for the effect of the rubber membrane if the error in principal stress difference due to the stiffness of the membrane exceeds 5 %:

$$\Delta(\sigma_1 - \sigma_3) = \frac{4E_m t \epsilon_1}{D}$$

where:

$\Delta(\sigma_1 - \sigma_3)$ = correction to be subtracted from the measured principal stress difference.

D = $\sqrt{\frac{4A}{\pi}}$ = diameter of specimen.

E_m = Young's modulus for the membrane material.

t = thickness of the membrane, and

ϵ_1 = axial strain.

8.5.1 The Young's modulus of the membrane material may be determined by hanging a 10.0-mm wide strip of membrane over a thin rod, placing another rod along the bottom of the hanging membrane, and measuring the force per unit strain obtained by stretching the membrane. The modulus value may be computed using the following equation assuming units are consistent:

$$E_m = \frac{FL}{A_m \Delta L}$$

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where:

E_m = Young's modulus of the membrane material,
F = force applied to stretch the membrane,
A_m = twice the initial thickness of the membrane multiplied by the width of the membrane strip,
L = unstretched length of the membrane, and
ΔL = change in length of the membrane due to application of F.

A typical value of E_m for latex membrane is 1400 kN/m^2.

NOTE 16—The effect of the stiffness of the membrane on the lateral stress is usually assumed to be negligible.

NOTE 17—The correction for rubber membranes is based on simplified assumptions concerning their behavior during shear. Their actual behavior is complex and there is not a consensus on more exact corrections.

8.6 Calculate the major and minor principal total stresses at failure as follows:

σ_3 = minor principal total stress = chamber pressure, and
σ_1 = major principal total stress = deviator stress at failure plus chamber pressure.

8.7 Calculate the initial degree of saturation of the test specimen using the initial mass and dimensions.

NOTE 18—The specific gravity determined in accordance with Test Method D 854 is required for calculation of the degree of saturation, or an assumed value may be used provided it is noted in the test report that an assumed value was used.

9. Report

9.1 The report shall include the following:

9.1.1 Identification and visual description of specimen, including soil group name, symbol, whether specimen is undisturbed, remolded, or compacted, and the like. Also include specimen identifying information, such as project, location, boring number, sample number, depth, and the like. Visual descriptions shall be made in accordance with Practice D 2488.

9.1.2 Initial dry unit weight and water content (specify if the water content specimen was obtained before or after the shear and from cuttings or the entire specimen).

9.1.3 Degree of saturation.

9.1.4 Height and diameter of the specimen.

9.1.5 Height to diameter ratio.

9.1.6 The value of the compressive strength and the values of the minor and major principal stresses at failure.

9.1.7 Stress - strain curve as described in 8.4.

9.1.8 Axial strain at failure, in percent.

9.1.9 Average rate of axial strain to failure, percent per minute.

9.1.10 Liquid and plastic limits, if determined, in accordance with Test Method D 4318.

9.1.11 Sketch or photo showing type of failure, that is, bulge, diagonal shear, and the like.

9.1.12 Particle-size analysis, if determined, in accordance with Method D 422.

9.1.13 If a membrane correction was used, the report shall state that a membrane correction was used to adjust the compressive strength and must indicate the membrane correction equation that was used.

9.1.14 In a remarks section note any unusual conditions or other data that would be considered necessary to properly interpret the results obtained, for example, slickensides, stratification, shells, pebbles, roots, or brittleness.

10. Precision and Bias

10.1 No method presently exists to evaluate the precision of a group of triaxial compression tests on undisturbed specimens, due to specimen variability. Undisturbed soil specimens from apparently homogeneous soil deposits at the same location often exhibit significantly different strength and stress - strain properties.

10.2 A suitable test material and method of specimen preparation have not been developed for the determination of laboratory variances of compacted specimens due to the difficulty in producing identical cohesive soil specimens. No estimates of precision for this test method are available.

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Designation: D 421 - 85

Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[1]

This standard is issued under the fixed designation D 421; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the dry preparation of soil samples as received from the field for particle-size analysis and the determination of the soil constants.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

3. Significance and Use

3.1 This practice can be used to prepare samples for particle-size and plasticity tests where it is desired to determine test values on air-dried samples, or where it is known that air drying does not have an effect on test results relative to samples prepared in accordance with Practice D 2217.

4. Apparatus

4.1 *Balance*, sensitive to 0.1 g.

4.2 *Mortar and Rubber-Covered Pestle*, suitable for breaking up the aggregations of soil particles.

4.3 *Sieves*—A series of sieves, of square mesh woven wire cloth, conforming to Specification E 11. The sieves required are as follows:

No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 40 (425-μm)

4.4 *Sampler*—A riffle sampler or sample splitter, for quartering the samples.

5. Sampling

5.1 Expose the soil sample as received from the field to the air at room temperature until dried thoroughly. Break up the aggregations thoroughly in the mortar with a rubber-covered pestle. Select a representative sample of the amount required to perform the desired tests by the method of quartering or by the use of a sampler. The amounts of material required to perform the individual tests are as follows:

5.1.1 *Particle-Size Analysis*—For the particle-size analysis material passing a No. 10 (2.00-mm) sieve is required in amounts equal to 115 g of sandy soils and 65 g of either silt or clay soils.

5.1.2 *Tests for Soil Constants*—For the tests for soil constants, material passing the No. 40 (425-μm) sieve is required in total amount of 220 g, allocated as follows:

Test	Grams
Liquid limit	100
Plastic limit	15
Centrifuge moisture equivalent	10
Volumetric shrinkage	30
Check tests	65

6. Preparation of Test Sample

6.1 Select that portion of the air-dried sample selected for purpose of tests and record the mass as the mass of the total test sample uncorrected for hygroscopic moisture. Separate the test sample by sieving with a No. 10 (2.00-mm) sieve. Grind that fraction retained on the No. 10 sieve in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains. Then separate the ground soil into two fractions by sieving with a No. 10 sieve.

6.2 Wash that fraction retained after the second sieving free of all fine material, dry, and weigh. Record this mass as the mass of coarse material. Sieve the coarse material, after being washed and dried, on the No. 4 (4.75-mm) sieve and record the mass retained on the No. 4 sieve.

7. Test Sample for Particle-Size Analysis

7.1 Thoroughly mix together the fractions passing the No. 10 (2.00-mm) sieve in both sieving operations, and by the method of quartering or the use of a sampler, select a portion weighing approximately 115 g for sandy soils and approximately 65 g for silt and clay soil for particle-size analysis.

8. Test Sample for Soil Constants

8.1 Separate the remaining portion of the material passing the No. 10 (2.00-mm) sieve into two parts by means of a No. 40 (425-μm) sieve. Discard the fraction retained on the No.

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[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved July 26, 1985. Published September 1985. Originally published as D 421 - 35 T. Last previous edition D 421 - 58 (1978)[ε1].

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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40 sieve. Use the fraction passing the No. 40 sieve for the determination of the soil constants.

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Designation: D 422 - 63 (Reapproved 1972)[ε1]

Standard Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[ε1] NOTE—Section 2 was added editorially and subsequent sections renumbered in July 1984.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-μm), or No. 200 (75-μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending on soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm.

2. Referenced Documents

2.1 *ASTM Standards:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Present edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 - 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.01.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.

ATTACHMENT - 3

9. Analytical Results

Triaxial Shear

- specimen description
- initial dry unit weight + water content, when water content obtained
- degree of saturation
- height + diameter of specimen, height to diameter ratio
- value of compressive strength, values of minor + major principal stresses at failure
- stress-strain curve
- axial strain at failure (percent)
- average rate of axial strain to failure (percent per minute)
- sketch or photo showing type of failure
- membrane correction equation used (if applicable

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ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

Geotech-9
W-20-50

Designation: D 2850 – 87

AMERICAN SOCIETY FOR TESTING AND MATERIALS
1916 Race St., Philadelphia, Pa 19103

Standard Test Method for UNCONSOLIDATED, UNDRAINED COMPRESSIVE STRENGTH OF COHESIVE SOILS IN TRIAXIAL COMPRESSION[1]

This standard is issued under the fixed designation D 2850; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the unconsolidated, undrained compressive strength (or maximum principal stress difference) of cylindrical specimens of cohesive soils in undisturbed, remolded, or compacted conditions using constant rate of deformation (strain-controlled) application of the axial compression test load and where the specimen is subjected to a confining fluid pressure in a triaxial chamber. No drainage of the specimen is permitted during the test. The test method provides for the measurement of the total stresses applied to the specimen, that is, the stresses are not corrected for pore-water pressure. The total stress is the sum of the effective stress and the pore pressure.

1.2 This test method provides data for determining undrained strength properties and stress-strain relations for soils.

NOTE 1—The determination of the unconsolidated, undrained strength of cohesive soils without lateral confinement is covered by Test Methods D 2166.

NOTE 2—This test method does not provide a procedure for back pressure saturation of the test specimens. If back pressure saturation of the specimens is required, the test must be performed utilizing procedures and apparatus similar to those required for a consolidated undrained triaxial test. However, due to consolidation, which could occur during the saturation phase, this modified procedure is not truly unconsolidated. A test method for the consolidated undrained triaxial test is currently under development in Subcommittee D18.05.

NOTE 3—This test method does not include a procedure for obtaining pore pressure measurements. Furthermore, at the rapid strain rates used in this test method such measurements could be inaccurate. If pore pressure measurements are desired, alternative procedures such as the U.S. Bureau of Reclamation Method E-17 can be used.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]

D 653 Terms and Symbols Relating to Soil and Rock[2]

D 854 Test Method for Specific Gravity of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2166 Test Methods for Unconfined Compressive Strength of Cohesive Soil[2]

D 2216 Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures[2]

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.05 on Structural Properties of Soils.

Current edition approved April 24, 1987. Published June 1987. Originally published as D 2850 – 70. Last previous edition D 2850 – 82.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

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D4220 Practices for Preserving and Transporting Soil Samples[2]
D4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *triaxial compression test*—a test in which a cylindrical specimen of soil encased in an impervious membrane is subjected to a confining pressure and then loaded axially to failure in compression (as defined in 3.2.1).

3.1.2 *principal stress difference or deviator stress*—the difference between the major and minor principal stresses in a triaxial test.

3.1.2.1 *Discussion*—The principal stress difference or deviator stress is equal to the axial load applied to the specimen divided by the corrected cross-sectional area of the specimen, as prescribed in Section 8. The major principal stress in the specimen is equal to the deviator stress plus the chamber pressure, and the minor principal stress in the specimen is equal to the chamber pressure.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *failure*—the failure stresses are taken as the stresses in the specimen corresponding to the maximum principal stress difference (deviator stress) attained or the principal stress difference (deviator stress) at 15 % axial strain, whichever is obtained first during the performance of a test.

3.2.2 *unconsolidated-undrained compressive strength*—the value of the maximum principal stress difference or deviator stress obtained during the test.

4. Significance and Use

4.1 In this test method, the compressive strength of a soil is determined in terms of the total stress, therefore, the resulting strength depends on the pressure developed in the pore fluid during loading. In this test method, fluid flow is not permitted from or into the soil specimen as the load is applied, therefore the resulting pore pressure, and hence strength, differs from that developed in the case where drainage can occur.

4.2 If the test specimens are 100 % saturated, consolidation cannot occur when the confining pressure is applied nor during the shear portion of the test since drainage is not permitted. Therefore, if several specimens of the same material are tested, and if they are all at approximately the same water content and void ratio when they are tested, they will have approximately the same undrained shear strength. The Mohr failure envelope will usually be a horizontal straight line over the entire range of confining stresses applied to the specimens if the specimens are fully saturated.

4.3 If the test specimens are partially saturated or compacted specimens, where the degree of saturation is less than 100 %, consolidation may occur when the confining pressure is applied and during shear, even though drainage is not permitted. Therefore, if several partially saturated specimens of the same material are tested at different confining stresses, they will not have the same undrained shear strength. Thus, the Mohr failure envelope for unconsolidated undrained triaxial tests on partially saturated soils is usually curved.

4.4 The unconsolidated undrained triaxial strength is applicable to certain design situations in geotechnical engineering practice where the loads are assumed to take place so rapidly that there is insufficient time for the induced pore-water pressure to dissipate and for consolidation to occur during the loading period (that is, drainage does not occur). The unconsolidated undrained triaxial strength is used to determine strengths at the end of construction.

4.5 Compressive strengths determined using this procedure may not apply in cases where the loading conditions in the field differ significantly from those used in this test method.

5. Apparatus

5.1 *Axial Loading Device*—The axial compression device may be screw jack driven by an electric motor through a geared transmission, a hydraulic or pneumatic loading device, or any other compression device with sufficient capacity and control to provide the rate of loading prescribed in 7.5. When the loading device is set to advance at a certain rate of strain, the actual rate of strain shall not deviate by more than ±10 %. Vibrations due to the operation of the loading device shall be kept at a minimum.

NOTE 4—A loading device may be said to provide sufficiently small vibrations if there are no visible ripples in a glass of water placed on the loading platen when the device is operating at the speed at which the test is performed.

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5.2 *Axial Load-Measuring Device*—The axial load-measuring device shall be a load ring, electronic load cell, hydraulic load cell, or any other load-measuring device capable of the accuracy prescribed in this section and may be a part of the axial loading device. The axial load-measuring device shall be capable of measuring the axial load to an accuracy of 1 % of the estimated axial load at failure.

5.3 *Chamber Pressure-Maintaining and Measurement Device*—The chamber pressure-maintaining and measurement device shall be capable of applying and controlling the chamber pressure to within ±1 % of the applied chamber pressure. This device may consist of a reservoir connected to the triaxial chamber and partially filled with the chamber fluid (usually water), with the upper part of the reservoir connected to a compressed gas supply; the gas pressure being controlled by a pressure regulator and measured by a pressure gage, electronic pressure transducer, or any other device capable of measuring to the prescribed tolerance. However, a hydraulic system pressurized by deadweight acting on a piston or any other pressure-maintaining and measurement device capable of applying and controlling the chamber pressure to the tolerance prescribed in this section may be used.

5.4 *Triaxial Compression Chamber*—An apparatus shall be provided in which the cylindrical specimen, enclosed by a membrane sealed to the specimen cap and base, may be placed and subjected to a constant hydrostatic fluid pressure. The apparatus shall include a bushing and piston, aligned with the axis of the specimen, through which the load from the axial loading device is transmitted to the specimen axially between the specimen cap and base. The bushing and piston shall be designed to minimize friction and lateral thrust to the specimen cap.

5.5 *Specimen Cap and Base*—An impermeable rigid cap and base shall be used to prevent drainage of the specimen. The specimen cap and base shall be constructed of a noncorrosive impermeable material, and each shall have a circular plane surface of contact with the specimen and a circular cross section. The weight of the specimen cap shall produce an axial stress on the specimen of less than 1 kN/m^2. The diameter of the cap and base shall be equal to the initial diameter of the specimen. The specimen base shall be coupled to the triaxial compression chamber so as to prevent lateral motion or tilting and the specimen cap shall be designed to receive the piston such that the piston-to-cap contact area is concentric with the cap. The specimen cap during shear shall not tilt more than 5°. The cylindrical surface of the specimen base and cap that contacts the membrane to form a seal shall be smooth and free of scratches.

NOTE 5—The stress produced by the specimen cap can exceed 1 kN/m^2 provided the test data is corrected for the effects of that stress.

5.6 *Deformation Indicator*—The deformation indicator shall be a dial indicator capable of measuring axial deformation to within 0.03 % of the specimen height and having a travel range of at least 20 % of the initial height of the test specimen, or any other measuring device, such as electronic deformation measuring devices, meeting these requirements of readability and range.

5.7 *Rubber Membranes*—The rubber membrane used to encase the specimen shall provide reliable protection against leakage. Membranes shall be carefully inspected prior to use, and if any flaws or pinholes are evident, the membrane shall be discarded. In order to offer minimum restraint to the specimen, the unstretched membrane diameter shall be between 90 and 95 % of that of the specimen. The membrane thickness shall not exceed 1 % of the diameter of the specimen. The membrane shall be sealed to the specimen base and cap by any method that will produce a positive seal. An equation for correcting the principal stress difference (deviator stress) for the effect of the stiffness of the membrane is given in 8.5.

NOTE 6—The membrane is typically sealed using O-rings with silicon grease between the cap and base and the membrane.

5.8 *Sample Extruder*—The sample extruder shall be capable of extruding the soil core from the sampling tube in the same direction of travel in which the sample entered the tube and with minimum disturbance of the sample. If the soil core is not extruded vertically, care should be taken to avoid bending stresses on the core due to gravity. Conditions at the time of sample removal may dictate the direction of removal, but the principal concern is to keep the degree of disturbance minimal.

5.9 *Specimen Size Measurement Devices*—Devices used to measure the height and diameter

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of the specimen shall be capable of measuring the desired dimension to within 0.1 % of its actual length and shall be constructed such that their use will not disturb the specimen.

5.10 *Timer*—A timing device indicating the elapsed testing time to the nearest 1 s shall be used for establishing the rate of strain application prescribed in 7.5.

5.11 *Balances*—The balance used to weigh specimens shall determine the mass of the specimens to within 0.1 % of the total mass.

5.12 *Apparatus for Water Content*, as specified in Method D 2216.

5.13 *Miscellaneous Apparatus*—Specimen trimming and carving tools, membrane and O-ring expanders, compaction apparatus, and data sheets as required.

6. Test Specimens

6.1 *Specimen Size*—Specimens shall have a minimum diameter of 30 mm and the largest particle contained within the test specimen shall be smaller than 1/6 of the specimen diameter. If, after completion of a test, it is found that oversize particles are present, indicate this information in the report of test data under remarks. Determine the average height and diameter of the test specimen using the apparatus specified in 5.9. Take a minimum of three height measurements (120° apart) and at least three diameter measurements at each of the quarter points of the height. The height-to-diameter ratio of the specimen shall be between 2 and 2.5.

NOTE 7—If large soil particles are found in the specimen after testing, a particle-size analysis in accordance with Method D 422 may be performed to confirm the visual observation and the results provided with the test report.

6.2 *Undisturbed Specimens*—Prepare undisturbed specimens from large undisturbed samples or from samples secured in accordance with Practice D 1587 or other acceptable undisturbed tube sampling procedures. Undisturbed samples shall be preserved and transported as outlined for Groups C or D samples in Practices D 4220. Specimens obtained by tube sampling may be tested without trimming, except for the squaring of ends, provided soil characteristics are such that no significant disturbance results from sampling and the specimen is uniformly circular. Handle specimens carefully to minimize disturbance, changes in cross section, or loss of water content. If compression or any type of noticeable disturbance would be caused by the extrusion device, split the sample tube lengthwise or cut it off in small sections to facilitate removal of the specimen with minimum disturbance. Prepare trimmed specimens in an environment where the change in the water content of the soil is minimized (Note 8). Specimens shall be of uniform, circular cross section perpendicular to the axis of the specimen. Where pebbles or crumbling result in excessive irregularity along the outside edges of the specimen or at the ends, pack soil from the trimmings in the irregularities to produce the desired surface. As an alternative, the ends of the specimen may be capped with a minimal thickness of plaster of paris, hydrostone, or similar material. Where soil conditions permit, a vertical lathe accommodating the total sample may be used as an aid in trimming the specimen to the required diameter. Determine the mass and dimensions of the test specimen in accordance with 5.9 and 5.11. If the specimen is to be capped, determine its mass and dimensions before capping. Enclose the specimen in the rubber membrane and seal the membrane to the specimen base and cap immediately after preparation.

NOTE 8—A controlled high-humidity room is usually used for this purpose.

6.3 *Remolded Specimens*—Prepare the specimen by first thoroughly working the undisturbed specimen, which has been tested and is still encased in the rubber membrane, with the fingers. Then reform the specimen by forming within a mold having dimensions such that the remolded specimen dimensions will be equal to those of the undisturbed specimen. Exercise care to avoid entrapping air in the specimen. This will aid in obtaining a uniform unit weight, in remolding to the same void ratio as the undisturbed specimen, and in preserving the natural water content of the soil.

6.4 *Compacted Specimens*—Prepare specimens using the compaction method, predetermined water content, and unit weight prescribed by the individual assigning the test. Compacted specimens may be prepared by compacting material in at least six layers, using a pressing or kneading action, into a split mold of circular cross section having dimensions meeting the requirements of 6.1. Material required for the specimen shall be batched by thoroughly mixing soil with sufficient water to produce the desired water

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U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: = V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart IN

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:
physical soil tests on soil + sediment, low level
- grain size
- triaxial compression

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
21 low level soils/sediment for grain size
5 low level soils for triaxial compression

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):
Superfund RI

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4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment: overnight courier
expect all samples in one shipment

6. Number of days analysis and data required after laboratory receipt of samples:
30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): —
Particle size: ASTM D422, D421, D854 (attached)
Triaxial Compression: ASTM D2850 (attached)

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):
See Attachment 1 (particle size).
Use at least 10 readings to define the triaxial stress-strain curve.
USE ONLY THE METHODS SPECIFIED ABOVE, OBTAIN APPROVAL OF EPA REGION II PRIOR TO USE OF ANY OTHER METHOD.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.
See Attached Attachment 2 + Attachment 3

10. Other (use additional sheets or attach supplementary information, as needed):
LAB DATA WILL BE REJECTED AND NON-PAYMENT WILL BE RECOMMENDED IF LAB DOES NOT FOLLOW METHODS CITED ABOVE.
See Attachment 10

11. Name of sampling/shipping contact: Greg Ruechel
Phone: (914) 458-8711

8.(cont.) Sieve, hydrometer, specific gravity + hygroscopic moisture analyses MUST be performed.

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12. Data Requirements

Parameter	Detection Limit	Precision Desired (±% or Concentration)
grain size % finer or passing	1%	

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
grain size	lab duplicate 1 per 10 samples or less	coarse fraction (> No. 10) 35% RPD
		fine fraction (< No. 10) 20% RPD
		hydrometer each fraction < 10% RPD
triaxial compression	—	none

14. Action Required if Limits are Exceeded

Reanalyze, contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

Geotech-4

SAMPLE PREPARATION ATTACHMENT 1

1.) Air-dry entire sample (see below), record air-dried weight (A)
2.) If the sample contains large pieces of material other than soil such as wood, leaves, leather, mats of hair, etc., this material should be removed by the analyst after the original air-dried weight has been recorded. A short written description of the material removed should be recorded. Record weight of material removed (B).
3.) Record weight of air-dried soil submitted, (A) - (B).
NOTE: Air-dry sample by spreading out sample in an aluminum pan or a crucible to dry. Length of time to air-dry sample (24 hours to possibly several days) will depend on type of soil received and its moisture content. Final air-dried soil should have <5% moisture.

MINIMUM SAMPLE SIZES

The air-dried soil is prepared for mechanical analysis as described in ASTM D-421. The separation on the No. 10 sieve results in a coarse and a fine fraction.

ASTM D-422 gives minimum sample sizes for samples with nominal diameters of the largest particles ranging from 3/8" to 3". Furthermore, these samples as well as samples with the largest particles less than 3/8" require a minimum sample size to yield portions passing a No. 10 sieve of 115 grams for sandy soils and 65 grams for silt/clay soils.

It is acceptable to use entire sample for analysis to meet these minimum requirements.

If these minimum requirements are not possible due to insufficient sample size, OR if the total weight of air-dried soil submitted is less than 200 grams, CONTACT SMO IMMEDIATELY (SMO will contact the Region). Give SMO the following information for the samples not meeting the ASTM D-422 requirements or the 200 gram requirement: sample numbers, sample weights, and required sample weights. Region V CRL will determine whether to proceed with or terminate analysis.

COARSE FRACTION:

Perform a sieve analysis of the portion retained on the No. 10 sieve according to the ASTM test method using the following sieves: 3" (75 mm), 2" (50 mm), 1 1/2" (37.5 mm), 1" (25.0 mm), 3/4" (19.0 mm), 3/8" (9.5 mm), No. 4 (4.75 mm), No. 10 (2.00 mm).

FINE FRACTION:

Perform hydrometer, hygroscopic moisture, and specific gravity analysis on the fraction that passes a No. 10 sieve. Give a written description of the portion passing No. 10 sieve. If sandy, use 100 grams for hydrometer analysis, and if clay/silt, use 50 grams for hydrometer analysis.

Perform a sieve analysis of the portion that passes a No. 10 sieve according to ASTM D-422 using the following sieves:
No. 20, No. 40, No. 60, No. 80, No. 100, No. 200.

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ATTACHMENT 2

RAW DATA:

All hand-written raw data must be submitted, and should include the following:

1.) Total air-dried weight of sample submitted (A).
2.) Total air-dried weight of extraneous material removed (B).
3.) Total air-dried weight of soil submitted (A) - (B).
4.) Total weight of soil retained on a No. 10 sieve.
5.) Total weight of soil passing a No. 10 sieve.
6.) Complete coarse fraction sieve analysis.
7.) Complete hydrometer analysis including time, temperature, hydrometer readings, type of hydrometer, etc.
8.) Complete hygroscopic moisture analysis.
9.) Complete specific gravity analysis.
10.) Complete fine fraction sieve analysis.
11.) Written description of extraneous material removed (if any), and of material passing a No. 10 sieve (sand or clay/silt).

Attachment A is suggested to be used as a raw data form by the laboratory, or the laboratory may use their own raw data forms as long as all required deliverables are included.

CALCULATIONS:

All calculations may be performed using a computer generated spreadsheet, but both handwritten raw data AND computer spreadsheets must be submitted as case deliverables.

REPORT:

Report results as percent finer than the specified particle size. Present data as tabulated AND in the form of a grain-sized distribution curve on a semi-logarithmic chart with percent finer by weight plotted on the arithmetic scale and grain size plotted on the logarithmic scale.

Attachment A

Geotech-6

ASTM D-422 Particle Size Analysis of Soils

Laboratory: _____

Analyste: _____

EPA Sample No.: _____

Lab Sample No.: _____

Sample Preparation:

Total air-dried sample: _____ g
Total air-dried extraneous material: _____ g
Total air-dried soil: _____ g
Total weight soil retained No. 10 sieve: _____ g
Total weight soil passing No. 10 sieve: _____ g
% Retained No. 10 Sieve: _____ %
% Passing No. 10 Sieve: _____ %

Extraneous material description: _____
% passing No. 10 description : _____

Coarse Sieve Analysis:	Sieve Size	Weight Retained, g
Date: _____	3"	_____
	2"	_____
	1 1/2"	_____
	1"	_____
	3/4"	_____
	3/8"	_____
	No. 4	_____
	No.10	_____

Hydrometer Analysis: Date: _____ Type of hydrometer: _____

Time (min.)	Actual Time	Hydrometer Actual	Hydrometer Comp. Corr.	Corrected Hydrometer	Temperature °C
2	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____
15	_____	_____	_____	_____	_____
30	_____	_____	_____	_____	_____
60	_____	_____	_____	_____	_____
250	_____	_____	_____	_____	_____
1440	_____	_____	_____	_____	_____

Fine Sieve Analysis:	Sieve Size:	Weight Retained, g
Date: _____	No. 20	_____
	No. 40	_____
	No. 60	_____
	No. 80	_____
	No. 100	_____
	No. 200	_____

Hygroscopic Moisture Analysis:

Tare: _____ g
Sample Weight: _____ g
Tare + Dry Weight: _____ g
Dry Weight: _____ g
% Moisture: _____ %

Specific Gravity Analysis:

Pycnometer: _____ g
Pycnometer, Water: _____ g
Pycnometer, Water, Soil: _____ g
Temperature, °C: _____
Specific Gravity: _____

Comments:

Total Phenol - 7

6.2 Biological degradation is inhibited by the addition of H_2SO_4 to pH <4. Store at 4°C. The sample is stable for 7 days prior to extraction and 40 days after extraction.

7.0 PROCEDURE

7.1 Distillation:

7.1.1 Measure 500 mL of sample into a beaker. Lower the pH to approximately 4 with concentrated H_2SO_4 (1 mL/L), and transfer to the distillation apparatus.

7.1.2 Distill 450 mL of sample, stop the distillation, and when boiling ceases, add 50 mL of warm reagent water to the flask and resume distillation until 500 mL have been collected.

7.1.3 If the distillate is turbid, filter through a prewashed membrane filter.

7.2 Direct photometric method:

7.2.1 Using working solution A (Step 5.8), prepare the following standards in 100-mL Class A volumetric flasks. A minimum of a blank and three standards must be prepared:

Working Solution A (mL)	Concentration (µg/L)
0.0	0.0
0.5	50.0
1.0	100.0
2.0	200.0
5.0	500.0
8.0	800.0
10.0	1000.0

7.2.2 To 100 mL of distillate or to an aliquot diluted to 100 mL and/or standards, add 2 mL of buffer solution (Step 5.4) and mix. The pH of the sample and standards should be 10 ± 0.2.

7.2.3 Add 2.0 mL aminoantipyrine solution (Step 5.5) and mix.

7.2.4 Add 2.0 mL potassium ferricyanide solution (Step 5.6) and mix.

7.2.5 After 15 min., read absorbance at 510 nm.

7.3 Chloroform extraction method:

CAUTION: This method should be performed in a hood; chloroform is toxic.

7.3.1 Using working solution B (Step 5.9), prepare the following standards. Standards may be prepared by pipetting the required volumes into the separatory funnels and diluting to 500 mL with reagent water. A minimum of a blank and three standards must be prepared:

Total Phenol-8

Working Solution B (mL)	Concentration (µg/L)
0.0	0.0
3.0	6.0
5.0	10.0
10.0	20.0
20.0	40.0
25.0	50.0

7.3.2 Place 500 mL of distillate or an aliquot diluted to 500 mL in a separatory funnel. The sample should not contain more than 50 µg/L phenol.

7.3.3 To sample and standards add 10 mL of buffer solution (Step 5.4) and mix. The pH should be 10 ± 0.2.

7.3.4 Add 3.0 mL aminoantipyrine solution (Step 5.5) and mix.

7.3.5 Add 3.0 mL potassium ferricyanide solution (Step 5.6) and mix.

7.3.6 After 3 min, extract with 25 mL of chloroform (Step 5.10). Shake the separatory funnel at least 10 times, let $CHCl_3$ settle, shake again 10 times, and let chloroform settle again.

7.3.7 Filter chloroform extract through filter paper. Do not add more chloroform.

7.3.8 Read the absorbance of the samples and standards against the blank at 460 nm.

7.4 Calculation:

7.4.1 Prepare a standard curve by plotting the absorbance values of standards versus the corresponding phenol concentrations.

7.4.2 Obtain concentration value of sample directly from standard curve.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 After calibrating verify calibration with an independently prepared check standard.

Total Phenol-9

8.5 The matrix duplicate and matrix spike are brought through the whole sample preparation and analytical process.

9.0 METHOD PERFORMANCE

9.1 In a single laboratory using sewage samples at concentrations of 3.8, 15, 43, and and 89 µg/L, the standard deviations were ±0.5, ±0.6, ±0.6, and ±1.0 µg/L, respectively. At concentrations of 73, 146, 299, and 447 µg/L, the standard deviations were ±1.0, ±1.8, ±4.2, and ±5.3 µg/L, respectively.

9.2 In a single laboratory using sewage samples at concentrations of 5.3 and 82 µg/L, the recoveries were 78% and 98%, respectively. At concentrations of 168 and 489 µg/L, the recoveries were 97% and 98%, respectively.

10.0 REFERENCES

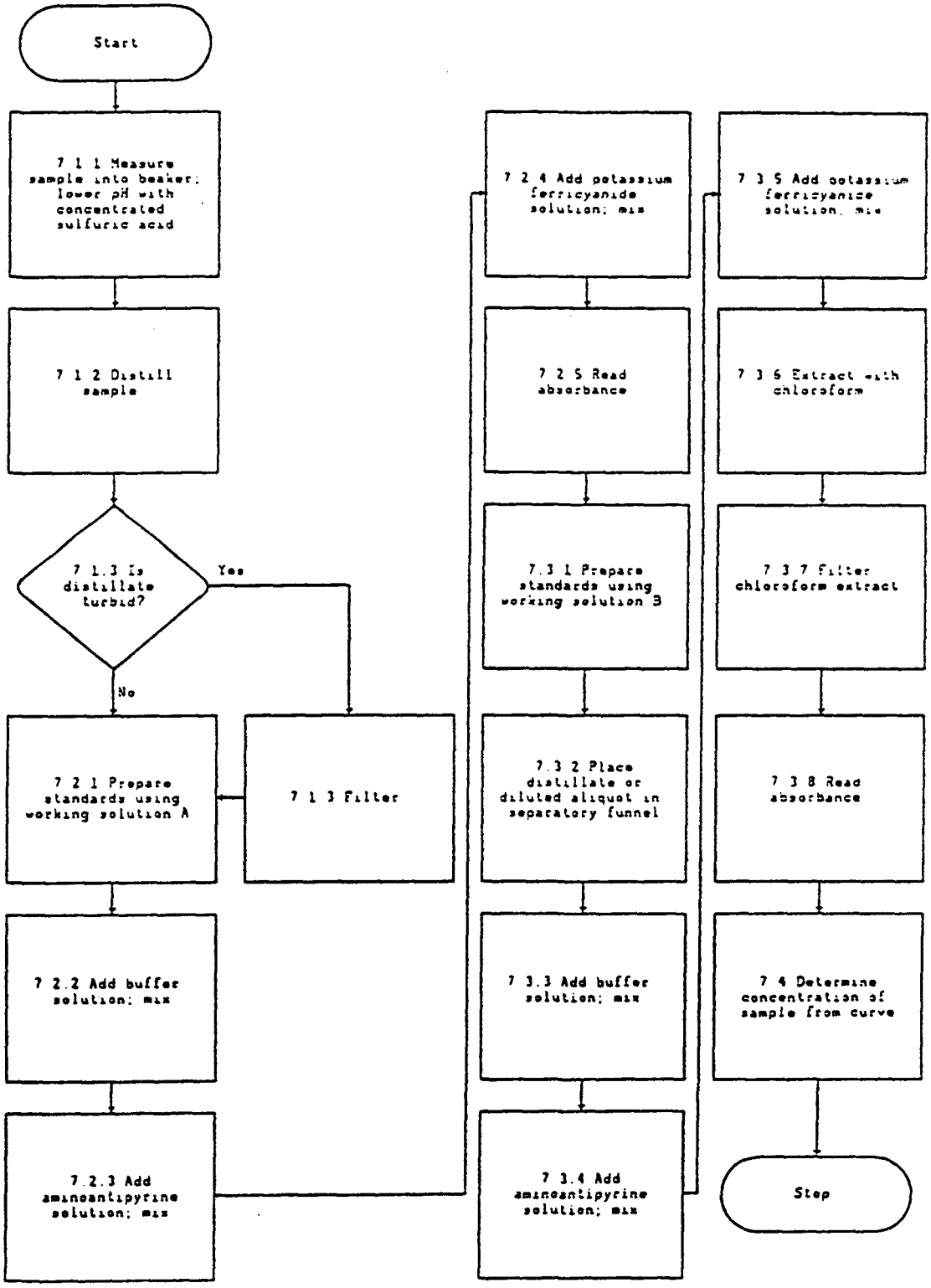
1. Annual Book of ASTM Standards, Part 31, "Water," Standard D1783-70, p. 553 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 14th ed., pp. 574-581, Method 510 through 510C (1975).

Total Phenol-TO

METHOD 9065A

PHENOLICS (SPECTROPHOTOMETRIC, MANUAL 4-AAP WITH DISTILLATION)



Total Phenol-11

Determine the standard deviation (s) for each analyte as follows:

$$s = (S^2)^{1/2}$$

Determine the MDL for each analyte as follows:

$$MDL = t_{(n-1,\ \alpha = .99)}(s)$$

where $t_{(n-1,\ \alpha = .99)}$ is the one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99 percent level.

ORGANIC-FREE REAGENT WATER:

For volatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water. Organic-free reagent water may also be prepared by boiling water for 15 minutes and, subsequently, while maintaining the temperature at 90°C, bubbling a contaminant-free inert gas through the water for 1 hour.

For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the highest of either:

(1)The detection limit, or

(2)Five percent of the regulatory limit for that analyte, or

(3)Five percent of the measured concentration in the sample.

For semivolatiles and nonvolatiles, all references to water in the methods refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. Organic-free reagent water can be generated by passing tap water through a carbon filter bed containing about 1 pound of activated carbon. A water purification system may be used to generate organic-free deionized water.

For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the highest of either:

(1)The detection limit, or

Total Phenol -12

(2)Five percent of the regulatory limit for that analyte, or

(3)Five percent of the measured concentration in the sample.

PRECISION: The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses. These samples should contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used estimates of precision are the relative standard deviation (RSD) or the coefficient of variation (CV),

$$RSD = CV = 100\ S/\bar{x},$$

where $\bar{x}$ = the arithmetic mean of the x_i measurements, and s = variance; and the relative percent difference (RPD) when only two samples are available.

$$RPD = 100\ [(x_1 - x_2)/((x_1 + x_2)/2)].$$

PROJECT: Single or multiple data collection activities that are related through the same planning sequence.

QUALITY ASSURANCE PROJECT PLAN (QAPJP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

RCRA: The Resource Conservation and Recovery Act.

REAGENT BLANK: See Method Blank.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For a method blank to be acceptable for use with the accompanying samples, the concentration in the blank of any analyte of concern must be no higher than the highest of either:

(1)The detection limit, or

(2)Five percent of the regulatory limit for that analyte, or

(3)Five percent of the measured concentration in the sample.

Total Phenol-13

For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

SPLIT SAMPLES: Aliquots of sample taken from the same container and analyzed independently. In cases where aliquots of samples are impossible to obtain, field duplicate samples must be taken for the matrix duplicate analysis. These are usually taken after mixing or compositing and are used to document intra-or interlaboratory precision.

STANDARD ADDITION: The practice of adding a known amount of an analyte to a sample immediately prior to analysis. It is typically used to evaluate interferences.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

SURROGATE: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

TRIP BLANK: A sample of media taken from the laboratory to the sampling site and returned to the laboratory unopened. The media used for the trip blank is acceptable if the concentration of any analyte of concern in the media is no higher than the highest of either:

(1)The detection limit, or

(2)Five percent of the regulatory limit for that analyte, or

(3)Five percent of the measured concentration in the sample.

A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

Total Phenol-5

METHOD 9065A

PHENOLICS (SPECTROPHOTOMETRIC, MANUAL 4-AAP WITH DISTILLATION)

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the analysis of ground water, drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 The method is capable of measuring phenolic materials at the 5 μg/L level when the colored end product is extracted and concentrated in a solvent phase using phenol as a standard.

1.3 The method is capable of measuring phenolic materials that contain more than 50 μg/L in the aqueous phase (without solvent extraction) using phenol as a standard.

1.4 It is not possible to use this method to differentiate between different kinds of phenols.

2.0 SUMMARY OF METHOD

2.1 Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown antipyrine dye. The amount of color produced is a function of the concentration of phenolic material.

3.0 INTERFERENCES

3.1 For most samples a preliminary distillation is required to remove interfering materials.

3.2 Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic-type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

3.3 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of <4 with H_2SO_4 and aerating briefly by stirring.

4.0 APPARATUS AND MATERIALS

4.1 Distillation apparatus: All glass, consisting of a 1-liter Pyrex distilling apparatus with Graham condenser.

4.2 pH meter.

4.3 Spectrophotometer: For use at 460 or 510 nm.

4.4 Funnels.

Total Phenol-6

4.5 Filter paper.

4.6 Membrane filters.

4.7 Separatory funnels: 500- or 1,000-mL.

4.8 Nessler tubes: Short or long form.

4.9 Class A volumetric flasks: 100 mL

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One. See Total Phenol-11

5.3 Sulfuric acid solution, H_2SO_4: Concentrated.

5.4 Buffer solution: Dissolve 16.9 g NH_4Cl in 143 mL concentrated NH_4OH and dilute to 250 mL with reagent water. Two mL of buffer should adjust 100 mL of distillate to pH 10.

5.5 Aminoantipyrine solution: Dissolve 2 g of 4-aminoantipyrine (4-AAP) in reagent water and dilute to 100 mL.

5.6 Potassium ferricyanide solution: Dissolve 8 g of $K_3Fe(CN)_6$ in reagent water and dilute to 100 mL.

5.7 Stock phenol solution: Dissolve 1.0 g phenol in freshly boiled and cooled reagent water and dilute to 1 liter (1 mL = 1 mg phenol).

NOTE: This solution is hydroscopic and toxic.

5.8 Working solution A: Dilute 10 mL stock phenol solution to 1 liter with reagent water (1 mL = 10 μg phenol).

5.9 Working solution B: Dilute 100 mL of working solution A to 1,000 mL with reagent water (1 mL = 1 μg phenol).

5.10 Chloroform.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

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U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES

Client Request

☒ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: = V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart IN

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:
determination of total phenol by distillation + reaction of distillate with alkaline ferricyanide + 4-AAP, measured colorimetrically

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):
5 low level aqueous samples

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.): Superfund Remedial

Total Phenol-2

4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment: daily by overnight carrier

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): —
EPA SW 846 Method 9066A

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. report check standard recovery, matrix spike (MS) recovery, lab duplicate (matrix spike) results & RPD, calibration curve correlation coefficient, lab blank concentration

10. Other (use additional sheets or attach supplementary information, as needed):
See Attachment 10

11. Name of sampling/shipping contact: Greg Ruechel
Phone: (414) 458-8711

Total Phenol-3

12. Data Requirements

Parameter	Detection Limit	Precision Desired (±% or Concentration)
Total phenol	2 ug/L	± 2 ug/L

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Lab blank (distilled)	1 per 10 samples or less	< 4 ug/L
~~Lab~~ matrix spike		75 - 125 %
matrix spike duplicate		75 - 125 %, 25 % RPD or < 4 ug/L
check standard		85 - 115 %

14. Action Required if Limits are Exceeded

Take corrective action + contact SMO

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

Total Phenol-4

ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

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eluent and separated anions to their respective acid forms.

4.2 For additional information, refer to the different manufacturers' instruction manuals.

5. Reagents

5.1 *Eluent*, 0.003 *M* sodium bicarbonate-0.0024 *M* sodium carbonate: Dissolve 0.2520 g $NaHCO_3$ and 0.2544 g Na_2CO_3 in demineralized water and dilute to 1 L (NOTE 2).

NOTE 2: Eluent concentration may be varied slightly to obtain the same retention times for each anion when a new separator column is used. The $NaHCO_3$ is subject to thermal decomposition and must be weighed without prior drying.

5.2 *Suppressor regeneration solution*, 1*N* H_2SO_4: *Cautiously* add 111 mL concentrated H_2SO_4 (sp gr 1.84) to approx 600 mL demineralized water. Cool and dilute to 4 L with demineralized water.

5.3 *Standard anion solutions*: Dry all salts for 1 h at 105°C unless otherwise specified. Store each standard solution in TFE-fluorocarbon bottles.

5.3.1 *Bromide standard solution*, 1.00 mL = 1.00 mg Br: Dissolve 1.2877 g NaBr in demineralized water and dilute to 1,000 mL.

5.3.2 *Chloride standard solution*, 1.00 mL = 1.00 mg Cl: Dissolve 1.6484 g NaCl in demineralized water and dilute to 1,000 mL.

5.3.3 *Fluoride standard solution*, 1.00 mL = 1.00 mg F: Dissolve 2.2101 g NaF in demineralized water and dilute to 1,000 mL.

5.3.4 *Nitrate-nitrogen standard solution*, 1.00 mL = 1.00 mg NO_3-N: Dissolve 6.0681 g $NaNO_3$ in demineralized water and dilute to 1,000 mL.

5.3.5 *Nitrite-nitrogen standard solution*, 1.00 mL = 1.00 mg NO_2-N: Dissolve 4.9259 g $NaNO_2$ in demineralized water and dilute to 1,000 mL.

5.3.6 *Phosphorus standard solution*, 1.00 mL = 1.00 mg P: Dissolve 4.3936 g anhydrous KH_2PO_4 in demineralized water and dilute to 1,000 mL.

5.3.7 *Sulfate standard solution*, 1.00 mL = 1.00 mg SO_4: Dissolve 1.8140 g K_2SO_4 in demineralized water and dilute to 1,000 mL.

5.4 *Mixed stock solution*: Prepare 1,000 mL mixed stock solution by appropriate quantitative dilution of each standard solution (NOTES 3 and 4).

Anion	Concentration (mg/L)	Volume (mL)
F	5.00	5
Cl	50.0	50
NO_2-N	5.0	5
PO_4-P	5.0	55
Br	5.0	5
NO_3-N	50.0	50
SO_4	50.0	50

NOTE 3. If nitrite is omitted from the mixed stock solution, the solution is stable for at least 1 month when stored and refrigerated in a clean TFE-fluorocarbon bottle. If nitrite is included in the mixed-stock solution, the solution needs to be prepared fresh daily.

NOTE 4. The above is only an example of a mixed-stock solution. Other appropriate concentrations can be prepared.

5.5 *Mixed standard solutions*: Prepare at least three mixed standard solutions by appropriate dilution of the mixed stock solution. The solutions should bracket the concentration range of interest.

6. Procedure

6.1 Set up the ion chromatograph according to the operating parameters described in 4.1. Equilibrate the columns with eluent until a stable baseline is obtained. Allow approximately 30 min for equilibration.

6.2 Set the full-scale conductivity to 10, 30, or 100 μS as is appropriate for the expected sample-anion concentrations. The higher settings are required for higher sample-anion concentrations.

6.3 Level the integrator at 10 mV (a display of 1000 with no signal). Adjust the ion chromatograph's offset to approximately 11 mV (a display of 1100). This ensures that the ion chromatograph's signal will not fall below 10 mV during the course of the analyses. The baseline signal tends to drift in a negative direction over a long period of time. Each chromatogram can be started at a signal level of 10 mV using the integrator's automatic-zero control.

6.4 Enter an appropriate program into the main program controller of the ion chromatograph according to the manufacturer's instruction manual. The system is configured so that the ion chromatograph controls the autosampler and starts the integrator at the beginning of each sample injection (NOTE 5).

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NOTE 5. For additional information on computerized data reduction, see Hedley and Fishman (1982).

6.5 Place the mixed standard solutions in the first positions of the sample tray followed by a standard reference material and then the samples. Place a standard reference material in every twentieth position of the remainder of the sample tray.

6.6 Create an information file in the integrator by pressing the DIALOG key. Through this information file, various integrator functions can be enabled or disabled during the recording of a chromatogram. The only necessary function is ER (end run). It terminates the chromatogram at the appropriate time as determined by the operator's setting of the ion chromatograph's controller, which actuates the sampler and causes the injection of a new sample.

6.7 Press the integrator's PT EVAL key before starting a series of analyses. The integrator will take about 50 s to store the baseline signal so that a peak can be distinguished from baseline noise. The baseline noise can be evaluated before each chromatogram, using the integrator's ET function.

6.8 Set the ion chromatograph's PGM/AUTO/MANUAL switch from MANUAL to AUTO and press Start/Step to begin the analyses.

7. Calculation

7.1 The integrator automatically computes the concentration of each anion in each sample by comparing its peak height or area to the analytical curve. Retention times for the seven anions are given in table 12.

8. Report

8.1 Report bromide (71870), chloride (00940), fluoride (00950), nitrate-nitrogen (00618), nitrite-nitrogen (00613), orthophosphate-phosphorus (00660), and sulfate (00945), dissolved, concentrations as follows: less than 1 mg/L, nearest 0.01 mg/L; 1 mg/L and above, two significant figures.

9. Precision

9.1 Analysis of a number of test samples 10 times each by one operator resulted in mean values, standard deviations, and percent relative standard deviations as shown in table 13.

Table 12.—Approximate retention times of anions by ion chromatography

Constituent	Time (min)
Fluoride	2.2
Chloride	3.3
Nitrate-nitrogen	4.0
Orthophosphate-phosphorus	4.9
Bromide	6.5
Nitrate-nitrogen	7.5
Sulfate	8.6

Table 13.—Precision for ion chromatographic determination of anions

Constituent	Mean (mg/L)	Standard deviation (mg/L)	Relative standard deviation (percent)
Bromide	0.295	0.020	6.8
Chloride	.72	.04	5.6
Do.	1.71	.06	3.5
Do.	2.72	.24	8.8
Do.	5.84	.19	3.2
Do.	9.90	.39	3.9
Do.	58.6	.7	1.2
Do.	119	1.2	1.0
Fluoride	.018	.004	22.2
Do	.080	.010	12.5
Do	.79	.02	2.5
Do	.92	.01	1.1
Do.	2.02	.15	7.4
Nitrate-nitrogen	.12	.01	8.3
Do	.42	.051	1.9
Do	.70	.081	1.4
Do.	1.27	.05	3.9
Do.	5.26	.14	2.7
Nitrite-nitrogen	.03	.01	33.3
Orthophosphate-phosphorus	.273	.010	3.7
Sulfate	1.68	.05	3.0
Do.	3.88	.10	2.6
Do.	15.1	.80	5.3
Do.	62.1	.9	1.4
Do.	100	1.4	1.4
Do.	148	3	2.0

References

Fishman, M. J., and Pyen, G. S., 1979, Determination of selected anions in water by ion chromatography: U.S. Geological Survey Water-Resources Investigations, 79-101, 30 p.

Hedley, A. G., and Fishman, M. J., 1982, Automation of an ion chromatograph for precipitation analysis with computerized data reduction: U.S. Geological Survey Water-Resources Investigations, 81-78, 33 p.

Small, H., Stevens, T. S., Bauman, W. C., 1975, Novel ion exchange chromatographic method using conductimetric detection: Analytical Chemistry, v. 47, p. 1801-9.

5/025 -1-1/89 · Total suspended solids in water 6/29/87

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
Phone: ~~(703) 557-2490~~ or FTS-557-2490

VSS - 1

SAS Number

Approved for Scheduling

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region and Site Name: Region V
B. Regional Representative: Jan Pels
C. Telephone Number: (312) 353-2720
D. Data request:
E. Site Name: Homco Dump

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis for volatile suspended solids (550 °C) in water (surface waters, groundwater, drinking water, leachate, etc.) Results are reported as mg/l volatile suspended solids.

2. Definition and number of work units involved (specify whether whole samples or fractions; whetner organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

5 low level aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial

4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment: Daily by overnight carrier.

(TSS-2)

6. Approximate number of days results required after ~~lab~~ receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

1. EPA Method 160.2, 1983 ed., (Gravimetric, Dried at 103° - 105° C) using glass fiber filter discs without organic binder such as: Millipore AP-40, Reeve Angel 934-AH, Gelman A/E, or equivalent. Use only membrane filter apparatus with 47 mm diameter glass fiber filter and a coarse (40-60 micron) fritted disc filter support. The filter and support specifications are mandatory. Samples will be held at 4°C until sample analysis and validation of results are completed. Holding time is 7 days from date of sample collection.

2. EPA Method 160.4 - Ignition of residue from 160.2.

8. Specail technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

1. Sample aliquot volumes are selected on the basis of the following factors. a) During initial sample filtration, filtration rate should not drop rapidly, or require more than 5 minutes of filtration time. (Increase the filter area or decrease the sample volume as needed for sample reanalysis), b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200ml in volume, and c) Sample aliquots should not exceed 200ml in volume. 2. Duplicate sample aliquots will be filtered with 2 or more intervening samples. 3. Final residues are to be weighed either to constant weight pursuant to Section 7.6 of Method 160.1 (The final weight is to be used for calculations), or dried overnight (12 hours of drying time) with the single weight used for calculations. Constant weight is defined as less than 0.5 mg or less than 4% weight loss from the previous weight, whichever is smaller. 4. Use only the methodsspecified above in items 7 and 8.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Identify EPA QC reference sample lot numbers used and their true values and 95% confidence intervals. Bench records of tare weights, final weights, volumes filtered, blanks, duplicate samples, and reference samples (all in the order filtered) will be provided along with copies of worksheets used to calculate results. Dates and time of a) filtration of initial 100ml volume, b) determination of tare weights, c) sample filtration, and d) determination of constant residue weights will be part of bench records. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed): See Attachment 10

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414 458 8711

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

VSS-3

ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

 VSS-4

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Volatile Suspended Solids	2-3 mg/l for 200 ml	Difference in duplicate results shall not exceed 0.5 mg for duplicate aliquots filtered.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.	sample aliquot	

II. QUALITY CONTROL REQUIREMENTS Do not use designated field blanks for QA Audits.

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
1) Lab Duplicates (See item 8.3 on Page 2)	1 per group of 10 or fewer samples	less than 0.5 mg for residue ≤5 mg; less than 10% for sample residue ≥5 mg
2) Lab Blanks (200 ml aliquots)	1 per group or 10 or fewer samples	-0.5 to +0.5 mg.
~~3) 1 set of 2 EPA QC Residue Reference Samples-2 concentration levels~~ not applicable to VSS	~~1 per sample set~~	~~<5 mg/l error for concentrations < to 50 mg/l or < or = 10% for nominal concentrations > than 50 mg/l~~

* Alternate reference samples must be approbed by Region V RSCC prior to analysis.

III. *Action Required if Limits are Exceeded:

Take corrective action and reanalyze samples.

Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

VSS-5

RESIDUE, VOLATILE

Method 160.4 (Gravimetric, Ignition at 550°C)

STORET NO. Total 00505
Non-Filterable 00535
Filterable 00520

1. Scope and Application
   1.1 This method determines the weight of solid material combustible at 550°C.
   1.2 The test is useful in obtaining a rough approximation of the amount of organic matter present in the solid fraction of sewage, activated sludge, industrial wastes, or bottom sediments.
2. Summary of Method
   2.1 The residue obtained from the determination of ~~total, filterable or~~ non-filterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/l volatile ~~residue~~ suspended solids.
3. Comments
   3.1 The test is subject to many errors due to loss of water of crystallization, loss of volatile organic matter prior to combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion.
   3.2 The results should not be considered an accurate measure of organic carbon in the sample, but may be useful in the control of plant operations.
   3.3 The principal source of error in the determination is failure to obtain a representative sample.
4. Sample Handling and Preservation
   4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decompostion of solids is recommended.
5. Precision and Accuracy
   5.1 A collaborative study involving three laboratories examining four samples by means of ten replicates showed a standard deviation of ±11 mg/l at 170 mg/l volatile residue concentration.
6. Reference
   6.1 The procedure to be used for this determination is found in:
   Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 95, Method 208E, (1975).

Approved for NPDES
Issued 1971

VSS-6

208 A. Total Residue Dried at 103-105 C

1. General Discussion

a. Principle: A well mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103 to 105 C. The increase in weight over that of the empty dish represents the total residue, which is an arbitrary quantity defined by the procedure followed. The determined values may not check with the theoretical value for solids calculated from the chemical analysis of water. Approximate methods for correlating the chemical analysis with the residue are available.[1] Although the results may not represent the weight of actual dissolved and suspended solids in wastewater samples, the determination serves a useful purpose for plant control. In some instances, correlation may be improved by adding 1 *N* sodium hydroxide to wastewater samples with a pH below 4.3 and maintaining the pH of 4.3 during evaporation. Correct the final calculation for the added sodium.

b. Interferences: Exclude large, floating particles or submerged agglomerates of nonhomogeneous materials from the sample. Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.

2. Apparatus

B.2 for Filtration Apparatus

All of the apparatus listed in Secti 208 A.2 is required and in addition:

*a. Glass fiber filter disks**, without organic binder.

b. Filtration apparatus suitable for the type of filter disk selected.

1) *Filter holder:* Gooch crucible adapter or membrane filter funnel.

2) *Gooch crucible*, 25-ml capacity for 2.2-cm-size glass fiber filter.

c. Suction flask, 500-ml capacity.

a. Evaporating dishes: Dishes of 100-ml capacity made of the following materials:

1) Porcelain, 90-mm diam.

2) Platinum—Generally satisfactory for all purposes.

3) Vycor*

b. Muffle furnace for operation at 550±50 C.

c. Steam bath.

d. Drying oven, equipped with a thermostatic control capable of maintaining the temperature within a 2 C range.

e. Desiccator, provided with a desiccant containing a color indicator of moisture concentration.

f. Analytical balance, 200-g capacity, capable of weighing to 0.1 mg.

3. Procedure

a. Ignite the clean evaporating dish at 550±50 C for 1 hr in a muffle furnace.

b. Cool, desiccate, weigh, and store the dish in a desiccator until ready for use.

c. Transfer the filtered measured sample to the preweighed dish and evaporate to dryness on a steam bath or in a drying oven. Choose a sample volume that will yield a minimum residue of 25 mg to 250 mg. Estimate the volume from the conductivity. If necessary, add successive portions of sample to the same dish. When evaporating in a drying oven, lower the temperature to approximately 98 C to prevent boiling and splattering.

d. Dry the evaporated sample for at least 1 hr at 103 to 105 C.

e. Cool the dish in a desiccator and weigh.

f. Repeat the cycle of drying at 103 to 105 C, cooling, desiccating, and weighing until a constant weight is obtained, or until loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less.

4. Calculation

$$\text{mg/l total residue} = \frac{(A-B)\times 1{,}000}{\text{ml sample}}$$

where A = weight of sample + dish and B = weight of dish.

5. Precision and Accuracy

The precision of the method is about ±4 mg or ±5%. When the residue from a 50- to 100-ml sample of raw sewage was weighed, the standard deviation of the weighing was found to be 1.9 mg (n=3; 60×10), but the data are considered statistically unreliable because of sampling errors. On settled effluents, a standard deviation of 0.9 mg (n=1; 5×20) was found and is statistically reliable.

*A product of Corning Glass Works, Corning, N.Y.

VSS-7

208 E. Total Volatile and Fixed Residue at 550 C

1. General Discussion

The volatile and fixed components in the total residue of Method A may be determined by igniting the sample at 550±50 C. The determination is useful in the control of wastewater plant operation because it offers a rough approximation of the amount of organic matter present in the solid fraction of wastewater, activated sludge, industrial wastes, or bottom sediments. Because the result also may reflect loss of water of crystallization, loss of volatile organic matter before combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion, it may not yield an accurate measure of organic carbon.

2. Apparatus

See Sections 208 A.2 and 208 B.2.

3. Procedure

Ignite the residue produced by Method A to constant weight in a muffle furnace at a temperature of 550±50 C. Have the furnace up to temperature before inserting the sample. (Usually, 15 to 20 min ignition are required.) Allow the dish to cool partially in air until most of the heat has been dissipated and transfer to a desiccator for final cooling in a dry atmosphere. Do not overload the desiccator. Weigh the dish as soon as it has cooled completely. Report the loss of weight on ignition as total volatile residue and the weighed residue as total fixed residue.

4. Calculation

$$\text{mg/l volatile residue} = \frac{(A-B)\times 1.000}{\text{ml sample}}$$

$$\text{mg/l fixed residue} = \frac{(B-C)\times 1.000}{\text{ml sample}}$$

where A = weight of residue+dish before ignition, B = weight of residue+dish after ignition, and C = weight of dish.

5. Precision and Accuracy

Three laboratories examined four samples by means of 10 replicates with a standard deviation of ±11 mg/l at 170 mg/l volatile residue concentration.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records, tabulating titrant standardization, titration volumes for titration or sample blanks (2 or more in number), samples, and QA Audits will be provided for each method used. All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QA Audit results. Records of chloride analysis will be provided for any samples so specified on the RAS/SAS Traffic Report or SAS Packing List. Separate bench records will be provided for any COD determinations of high chloride samples (>2000 mg/l Cl) including weight of mercuric sulfate used, sample titration volume and titration blank volume for each sample type. EPA QC Reference samples, or any other reference samples, will be identified as to source lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis results will be provided for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414-458-8711

5/017__-0-7/87 BOD in Water and Wastewater 7/30/87

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818, Alexandria, Virginia 22313
PHONE: (703)/557-2490 or FTS/557-2490

BOD – SAS Number

Approved for Scheduling

SPECIAL ANALYTICAL SERVICES
Client Request

[X] Regional Transmittal [ ] Telephone Request

A. EPA Region/Client: Region V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart IN

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of biological oxygen demand (BOD) in water and wastewater. Samples will be unfiltered. Results are reported as mg/l oxygen.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

5 low level aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial

4. Estimated date(s) of collection: _____

5. Estimated date(s) and method of shipment: Daily by overnight carrier, no Friday shipment for Saturday delivery

6. Number of days analysis and data required after laboratory receipt of samples:

30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

BOD "Standard Methods for the Examination of Water and Wastewater" 15th or 16th Edition, Method 507. All samples will be seeded unless otherwise stated.

8. Special technical instruction (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Set-up 3 or more sample dilutions so that two or more sample dilutions overlap to result in a residual D.O. > or = to 1 mg/l and a D.O. depletion ≥ 2 mg/l. Measure the seed BOD using 2 or more dilutions (Section 5d). BOD results for 2 dilutions should agree within + or - 15%. Analyze unseeded dilution water blanks , and glucose-glutamic acid checks (Section 5b of Method 507), both in duplicate, in addition to sample dilutions. Determine the initial and final D.O. for each bottle. Store samples at 4°C until analysis. The holding time is not to exceed 48 hours from the time of the beginning of sample collection. Dilution water will be seeded so that calculated DO uptake from BOD of seed will be between 0.6 and 1.0 mg/l (Section 5d of Method 507). Do not use seeded blanks to estimate seed corrections. All procedures defined in the Method must be followed precisely. Check for interferences (Section 5e).

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

All measurements and calculations must be documented and submitted. Submit all raw data. Report initial and final D.O. from each bottle. Report BOD in mg/l for each bottle and the average of each fitting the depletion range listed above using calculations specified by "Standard Methods"(Section 6 of Method 507). Report results of duplicates, unseeded dilution water blank, BOD of seed, calculated DO uptake of seed in seeded dilution water, and glucose-glutamic acid check.
EPA QC reference samples, or any other reference sample or initial calibration verification, will be identified as to source, lot number, and sample number. Corresponding "true" or target values and associated 95% confidence limits for analysis will be provide for all reference samples used.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414 458 8711

BOD-3

I. DATA REQUIREMENTS

Parameter:	Detection Limit	Precision Desired (±% or Conc.)
BOD	2 mg/l	Differences in duplicate series of sample results shall not exceed 2 mg/l for concentrations less than 20mg/l.

II. QC REQUIREMENTS Do not use any field blanks for QA audits.

Audits Required	Frequency of Audits	Limits* (% or Conc.)
Glucose-Glutamic acid checks	1 pair per set of samples	160-240 mg/l
Duplicate (full dilution series)	at least 1 per group of 10 or fewer samples	+ or -(10% or 2 mg/l)
Unseeded Dilution Water Blanks	1 pair per set of samples, including 1 pair for each lot of dilution water	< or = to 0.2 mg/l
DO Uptake of seed in seeded dilution water (calculated)	calculated for each lot of seeded dilution water	0.6 to 1.0 mg/l
1 set of EPA QC Demand Reference Samples (if specified) Yes X No ___	1 set of 2 per sample set	75 - 125% Recovery

III. ACTION REQUIRED IF LIMITS ARE EXCEEDED:

Take corrective action and reanalyze samples - Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

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P_3 = uncorrected net purgeable organic Cl^-, μg Cl^-/L, and
V = volume of sample or standard purged, L.

Determine the linear regression of instrument calibration standard curves for each instrument configuration. Update this linear regression daily by including the standard points analyzed on that day. Calculate the corrected organic chloride concentration for each replicate of each sample by substituting the net organic chloride content (C_4 or P_3) of each sample replicate into the appropriate linear regression equation.

7. Bibliography

DRESSMAN, R. C. Total Organic Halide, Method 450.1—Interim. Drinking Water Research Div., Municipal Environmental Research Lab., U.S. Environmental Protection Agency, Cincinnati, Ohio.

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TAKAHASHI, Y., et al. 1980. Measurement of total organic halides (TOX) and purgeable organic halides (POX) in water using carbon adsorption and microcoulometric detection. Proc. Symp. on Chemistry and Chemical Analysis of Water and Waste Water Intended for Reuse, Houston, Tex. American Chemical Soc., Washington, D.C.

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507 OXYGEN DEMAND (BIOCHEMICAL)*

1. Discussion

The biochemical oxygen demand (BOD) determination is an empirical test in which standardized laboratory procedures are used to determine the relative oxygen requirements of wastewaters, effluents, and polluted waters. The test measures the oxygen required for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. It also may measure the oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand) unless their oxidation is prevented by an inhibitor.

The method consists of placing a sample in a full, airtight bottle and incubating the bottle under specified conditions for a specific time. Dissolved oxygen (DO) is measured initially and after incubation. The BOD is computed from the difference between initial and final DO.

The bottle size, incubation temperature, and incubation period are all specified. Most wastewaters contain more oxygen-demanding materials than the amount of DO available in air-saturated water. Therefore, it is necessary to dilute the sample before incubation to bring the oxygen demand and supply into appropriate balance. Because bacterial growth requires nutrients such as nitrogen, phosphorus, and trace metals, these are added to the dilution water, which is buffered to ensure that the pH of the incubated sample remains in a

*Approved by Standard Methods Committee, 1981.

Br-3

12. Data Requirements

Parameter	Detection Limit	Precision Desired (±% or Concentration)
Bromide	0.10 mg/L	± 10% for bromide concentrations ≥ 0.50 mg/L otherwise ± 0.10 mg/L for concentrations ≤ 0.5 mg/L

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Lab Blank	daily (at least 1 per 20 spls)	< 0.10 mg/L
Lab Control Sample	daily (at least 1 per 20 spls)	85-115% recovery
* matrix spike	one per 10 samples	85-115% recovery
lab duplicate	one per 10 samples	± 10% rpd
Calibration Verification Standard	daily (at least 1 per 20 samples)	90-110% recovery

14. Action Required if Limits are Exceeded

Take corrective action and reanalyze samples: contact Sample Management Office (SMO)

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

* matrix spike concentration to be ≥ 30% sample concentration.

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ATTACHMENT 10

The following apply to the SAS Request Form sections as noted.

Section 7. Laboratory data rejection and non-payment will be recommended if the laboratory uses methods other than those specified in this SAS request.

Section 9. All original tags, chain of custody forms, SAS packing lists, airbills, and original data must be submitted to the Region within the time frame listed in section 6, above.

Br-5

Anions, ion-exchange chromatographic, automated

Parameters and Codes:

Anions, dissolved, I-2057-85 (see below)

Parameter	Code	Parameter	Code
Bromide (mg/L as Br)	71870	Nitrite (mg/L as N)	00613
Chloride (mg/L as Cl)	00940	Orthophosphate (mg/L as P)	00671
Fluoride (mg/L as F)	00950	Sulfate (mg/L as SO_4)	00945
Nitrate (mg/L as N)	00618		

1. Application

1.1 This method may be used only for the determination of dissolved bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate in natural water. Table 11 shows approximate lower and upper concentration limits. Actual limits depend on many factors including the column age, which affects column resolution, the relative concentrations of closely eluting species, and the volume of the sample injected. Samples containing anion concentrations high enough to overload the column resins or interfere with closely eluting species need to be diluted or a sample loop smaller than the 200-μL sample loop specified in this method needs to be used. Sample dilution or use of smaller volumes will change the detection limits for all anions.

1.2 Analyses must be performed on filtered and unacidified samples.

1.3 The ion chromatographic (IC) technology is so new that instruments and associated data-processing equipment and software available on the commercial market are not standardized and operating conditions vary enormously. Until operating conditions of various manufacturers' instruments become more comparable and the equivalency of methods using those instruments is established by extensive testing, the IC method approved for U.S. Geological Survey use will specify instrument and associated software brands. This does not imply endorsement of one product over another, but rather, acknowledges that IC technology is rapidly changing and developing.

Table 11.—Working ranges of anions by ion chromatography

Constituent	Minimum concentration[1] (mg/L)	Maximum concentration (mg/L)
Fluoride	0.01	50
Chloride	.20	50
Nitrite-nitrogen	.02	70
Orthophosphate-phosphorus	.06	40
Bromide	.10	150
Nitrate-Nitrogen	.05	150
Sulfate	.20	100

[1]With a larger sample loop (for example, 800 μL), minimum concentration levels can be lowered.

2. Summary of method

2.1 A sample is injected into an ion chromatograph and is pumped through three different ion-exchange columns into a specific-conductivity detector. The first two columns, a precolumn and separator column, are packed with low-capacity anion exchanger. Ions are separated based on their affinity for the exchange sites of the resin. The last column is a suppressor column that contains cation-exchange resin in the hydrogen form. The suppressor column reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample into their corresponding acids. The separated anions in their acid form are measured using an electrical-conductivity cell. Anions are identified based

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on their retention times compared with known standards. Quantitation is accomplished by measuring the peak height or area and by comparing it with an analytical curve generated from known standards.

2.2 During analysis, the suppressor column will slowly be exhausted and, therefore, will need to be regenerated. Other suppressors, such as the hollow-fiber suppressor, which is continuously regenerated, may be used.

2.3 For additional information on ion chromatography, see Small and others (1975) and Fishman and Pyen (1979).

3. Interferences

3.1 Because bromide and nitrate elute very closely together, they potentially interfere with each other. Bromide-to-nitrate ratios should not exceed 1:10 or 10:1 if both ions are to be quantitated.

3.2 High levels of organic acids may be present in industrial and domestic wastes which may interfere with inorganic-anion analysis. Two common species, formate and acetate, elute between fluoride and chloride.

3.3 Water from the sample injection will cause a negative peak or dip in the chromatogram when it elutes, because its conductivity is less than that of the suppressed eluent. This dip usually occurs between F^{-1} and Cl^{-1}. Any peak of interest eluting near the water dip must be sufficiently resolved from the dip to be accurately quantitated. A method of eliminating the conductivity drop due to bicarbonate and carbonate is to introduce into the sample concentrations of bicarbonate and carbonate that closely approximate those of the eluent used for analysis. Adjustment of the sample background may be accomplished in two ways.

3.3.1 Dilute the sample with eluent if sample dilution is required prior to analysis.

3.3.2 A volume of 1.0 mL of a prepared eluent concentrate (a solution that is 100 times more concentrated than the eluent with respect to bicarbonate and carbonate ions) can be added per 100.0 mL of sample. CAUTION: Samples prepared in this manner have a pH of about 10 and will readily absorb carbon dioxide if left exposed to the atmosphere. The result will cause a positive-peak interference.

3.3.3 Standard solutions need to be prepared in the same manner as the samples. It is important to prepare a blank using demineralized water at eluent strength in bicarbonate and carbonate to indicate any interferences that may have been introduced by the sample-preparation technique.

3.4 Samples containing high concentrations of chloride or other anions may prevent resolution of closely eluting peaks. For example, the peak for 0.1 mg of bromide per liter in the presence of greater than 1,000 mg of chloride per liter is swamped by the chloride peak. Bromide begins to elute before the chloride peak completely returns to the baseline.

3.5 Unexpected, late-eluting peaks are a potential source of interference. A peak eluting about two minutes after sulfate, believed to be oxalate, has been observed in some precipitation samples.

4. Apparatus

4.1 *Ion Chromatograph, Dionex Model 12; auto-sampler, Gilson; integrator (NOTE 1), Spectra Physics* using the following operating conditions:

Sample loop --------	200 μL
Eluent flow rate -----	138 mL/h (30 percent of full capacity)
Sample pump flow rate	50 percent of full capacity
Specific conductance meter settings ----	10, 30, or 100 μS

NOTE 1. A dual pen recorder (1 V and 100 mV) may replace an integrator. The recorder should be capable of full-scale response in two seconds or less. A typical chart speed is 0.5 cm/min.

4.1.1 *Precolumn*, 4 × 50-mm, fast-run, anion-resin column (Dionex P/N 030831 or equivalent) placed before the separator column to protect the separator column from contamination by particulates or species strongly retained by the ion-exchange resin.

4.1.2 *Separator column*, 4 × 250-mm, fast-run, anion-separator column packed with low-capacity, pellicular, anion-exchange resin (Dionex P/N 030830 or equivalent) that is styrene divinylbenzene-based. This is suitable for resolving fluoride, chloride, nitrite, orthophosphate, bromide, nitrate, and sulfate.

4.1.3 *Suppressor column*, 6 × 250-mm, column-packed, with a high-capacity, column-exchange resin (Dowex 50W-X 16-H form resin or equivalent) that is capable of converting the

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range suitable for bacterial growth. Complete stabilization of a sample may require a period of incubation too long for practical purposes; therefore, 5 d has been accepted as the standard incubation period.

Measurements of BOD that include both carbonaceous oxygen demand and nitrogenous oxygen demand generally are not useful; therefore, where appropriate, an inhibiting chemical may be used to prevent ammonia oxidation.[1] With this technique carbonaceous and nitrogenous demands can be measured separately. The inclusion of ammonia in the dilution water demonstrates that there is no intent to include the oxygen demand of reduced nitrogen forms in the BOD test. If this ammonia were oxidized, errors would result because the oxygen use would not be due exclusively to pollutants in the sample.

The extent of oxidation of nitrogenous compounds during the 5-d incubation period depends on the presence of microorganisms capable of carrying out this oxidation. Such organisms usually are not present in raw sewage or primary effluent in sufficient numbers to oxidize significant quantities of reduced nitrogen forms in the 5-d BOD test. Currently, many biological treatment plant effluents contain significant numbers of nitrifying organisms. Because oxidation of nitrogenous compounds can occur in such samples, inhibition of nitrification is recommended for samples of secondary effluent, for samples seeded with secondary effluent, and for samples of polluted waters.

The method included here contains both a dilution water check (5*b*) and a dilution water blank (5*h*). The dilution water check is to determine the acceptability of a particular batch of dilution water before it is used for BOD analysis. Seeded dilution waters are checked further for acceptable quality by measuring their consumption of oxygen from a known organic mixture, usually glucose and glutamic acid (5*c*).

The dilution water blank, made at the same time that samples are analyzed, provides a further quality control on dilution water at the time of analysis as well as on the cleanliness of apparatus such as BOD bottles.

The procedure for determining immediate oxygen demand (IDOD) has been eliminated because: (*a*) it was not clear whether IDOD should be reported in 5-d BOD data; (*b*) the measurement was inaccurate because of the small differences between initial DO and DO after 15 min; (*c*) arbitrary selection of 15 min for measuring IDOD did not necessarily include all short-term oxygen-consuming reactions; and (*d*) the IDOD is, in some instances, an iodine demand (during the DO determination) rather than a true DO demand. The methods outlined here require determining initial DO immediately after making the dilution. In this way all oxygen uptake (including that occurring during the first 15 min) is included in the BOD measurement.

Although only the 5-d BOD is described here, many variations of oxygen demand measurements exist. These include using shorter and longer incubation periods, tests to determine rates of oxygen uptake, continuous oxygen uptake measurements by respirometric techniques, etc.

2. Sampling and Storage

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values. Minimize reduction of BOD by analyzing the sample promptly or by cooling it to near-freezing temperature during storage. However, even at low temperature, keep the holding time to a minimum. Warm the chilled samples to 20°C before analysis; some storage time can be used to accomplish this conveniently.

a. Grab samples: If analysis is begun within 2 h of collection, cooling is unnecessary. If analysis is not started within 2 h of sample collection, keep sample at or be-

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low 4°C from the time of collection. Begin analysis within 6 h of collection; when this is not possible because the sampling site is distant from the laboratory, store at or below 4°C and report length and temperature of storage with the results. In no case start analysis more than 24 h after grab sample collection. When samples are to be used for regulatory purposes make every effort to deliver samples for analysis within 6 h of collection.

b. Composite samples: Keep samples at or below 4°C during compositing. Limit compositing period to 24 h. Use the same criteria as for storage of grab samples, starting the measurement of holding time from the end of the compositing period. State storage time and conditions as part of the results.

3. Apparatus

a. Incubation bottles: 250- to 300-mL capacity, with ground-glass stoppers. Clean bottles with a detergent, rinse thoroughly, and drain before use. As a precaution against drawing air into the dilution bottle during incubation, use a water-seal. Obtain satisfactory water seals by inverting bottles in a water bath or adding water to the flared mouth of special BOD bottles. Place a paper or plastic cup or foil cap over the flared mouth of the bottle to reduce evaporation of the water seal during incubation.

b. Air incubator or water bath: Thermostatically controlled at 20 ± 1°C. Exclude all light to prevent possibility of photosynthetic production of DO.

4. Reagents

a. Phosphate buffer solution: Dissolve 8.5 g KH_2PO_4, 21.75 g K_2HPO_4, 33.4 g $Na_2HPO_4 \cdot 7H_2O$, and 1.7 g NH_4Cl in about 500 mL distilled water and dilute to 1 L. The pH should be 7.2 without further adjustment. Discard reagent (or any of the following reagents) if there is any sign of biological growth in the stock bottle.

b. Magnesium sulfate solution: Dissolve 22.5 g $MgSO_4 \cdot 7H_2O$ in distilled water and dilute to 1 L.

c. Calcium chloride solution: Dissolve 27.5 g $CaCl_2$ in distilled water and dilute to 1 L.

d. Ferric chloride solution: Dissolve 0.25 g $FeCl_3 \cdot 6H_2O$ in distilled water and dilute to 1 L.

e. Acid and alkali solutions, 1N: For neutralization of caustic or acidic waste samples.

f. Sodium sulfite solution, 0.025N: Dissolve 1.575 g Na_2SO_3 in 1000 mL distilled water. This solution is not stable; prepare daily.

g. Nitrification inhibitor: 2-chloro-6-(trichloro methyl) pyridine.†

h. Glucose-glutamic acid solution: Dry reagent-grade glucose and reagent-grade glutamic acid at 103°C for 1 h. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to 1 L. Prepare fresh immediately before use.

5. Procedure

a. Preparation of dilution water: Place desired volume of water in a suitable bottle and add 1 mL each of phosphate buffer, $MgSO_4$, $CaCl_2$, and $FeCl_3$ solutions/L of water. Seed dilution water, if desired, as described in 5*d*. Test and store dilution water as described in 5*b* and 5*c* so that water of assured quality always is on hand.

b. Dilution water check: Use this procedure as a rough check on quality of dilution water. If dilution water has not been stored for quality improvement, add sufficient seeding material to produce a DO uptake of 0.05 to 0.1 mg/L in 5 d at 20°C. Do not seed dilution water that has been stored for quality improvement. Incubate a BOD bottle full of dilution water for 5 d at 20°C. Determine initial and final DO as in 5*g* and 5*j*. The DO uptake in 5 d at

†Nitrification Inhibitor 2533, Hach Chemical Co., or equivalent.

20°C should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.

If the oxygen depletion of a candidate water exceeds 0.2 mg/L obtain a satisfactory water by improving purification or from another source. Alternatively, if nitrification inhibition is used, store the seeded dilution water at 20°C until the oxygen uptake is sufficiently reduced to meet the dilution water check criteria. Storage is not recommended when BODs are to be determined without nitrification inhibition because nitrifying organisms may develop during storage. Check stored dilution water to determine whether sufficient ammonia remains after storage.

Before use bring dilution water temperature to 20°C. Saturate with DO by shaking in a partially filled bottle or by aerating with filtered air. Alternatively, store in cotton-plugged bottles long enough for water to become saturated with DO. Protect water quality by using clean glassware, tubing, and bottles.

c. Glucose-glutamic acid check: Because the BOD test is a bioassay the results can be influenced greatly by the presence of toxicants or by use of a poor seeding material. Distilled waters frequently are contaminated with copper; some sewage seeds are relatively inactive. Low results always are obtained with such seeds and waters. Periodically check dilution water quality, seed effectiveness, and analytical technique by making BOD measurements on pure organic compounds. In general, for BOD determinations not requiring an adapted seed, use a mixture of 150 mg glucose/L and 150 mg glutamic acid/L as a "standard" check solution. Glucose has an exceptionally high and variable oxidation rate but when it is used with glutamic acid, the oxidation rate is stabilized and is similar to that obtained with many municipal wastes. Alternatively, if a particular wastewater contains an identifiable major constituent that contributes to the BOD, use this compound in place of the glucose-glutamic acid.

Determine the 5-d 20°C BOD of a 2% dilution of the glucose-glutamic acid standard check solution using the techniques outlined in *5d-j*. If the 5-d 20°C BOD value of the check is outside the range of 200 ± 37 mg/L, reject any BOD determinations made with the seed and dilution water and seek the cause of the problem.

d. Seeding: It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in the sample. Domestic wastewater, unchlorinated or otherwise-undisinfected effluents from biological waste treatment plants, and surface waters receiving wastewater discharges contain satisfactory microbial populations. Some samples do not contain a sufficient microbial population (for example some untreated industrial wastes, disinfected wastes, high-temperature wastes, or wastes with extreme pH values). For such wastes seed the dilution water by adding a population of microorganisms. The preferred seed is effluent from a biological treatment system processing the waste. Where this is not available, use supernatant from domestic wastewater after settling at 20°C for at least 1 h but no longer than 36 h.

Some samples may contain materials not degraded at normal rates by the microorganisms in settled domestic wastewater. Seed such samples with an adapted microbial population obtained from the undisinfected effluent of a biological process treating the waste. In the absence of such a facility, obtain seed from the receiving water below (preferably 3 to 8 km) the point of discharge. When such seed sources also are not available, develop an adapted seed in the laboratory by continuously aerating a sample of settled domestic wastewater and adding small daily increments of waste. Optionally use a soil suspension or activated sludge to obtain the initial microbial population. Determine the exist-

ence of a satisfactory population by testing the performance of the seed in BOD tests on the sample. BOD values that increase with time of adaptation to a steady high value indicate successful seed adaptation. In making tests, use enough seed to assure satisfactory numbers of microorganisms but not so much that the oxygen demand of the seed itself is a major part of the oxygen used during incubation.

Determine BOD of the seeding material as for any other sample. This is the seed control. From the value of the seed control and a knowledge of the seeding material dilution (in the dilution water) determine seed DO uptake. To determine a sample DO uptake subtract the seed DO uptake from the total DO uptake. The DO uptake of the seeded dilution water should be between 0.6 and 1.0 mg/L.

Techniques for adding seeding material to dilution water are described for two sample dilution methods (¶ 5*f*).

e. Sample pretreatment:

1) Samples containing caustic alkalinity or acidity—Neutralize samples to pH 6.5 to 7.5 with a solution of sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH) of such strength that the quantity of reagent does not dilute the sample by more than 0.5%. The pH of seeded dilution water should not be affected by the lowest sample dilution.

2) Samples containing residual chlorine compounds—If possible, avoid samples containing residual chlorine by sampling ahead of chlorination processes. If the sample has been chlorinated but no detectable chlorine residual is present, seed the dilution water. If residual chlorine is present, dechlorinate and seed the dilution water (5*f*). Do not test chlorinated/dechlorinated samples without seeding the dilution water. In some samples chlorine will dissipate within 1 to 2 h of standing in the light. This often occurs during sample transport and handling. For samples in which chlorine residual does not dissipate in a reasonably short time, destroy chlorine residual by adding Na_2SO_3 solution. Determine required volume of Na_2SO_3 solution on a 100- to 1000-mL portion of neutralized sample by adding 10 mL of 1 + 1 acetic acid or 1 + 50 H_2SO_4, 10 mL potassium iodide (KI) solution (10 g/100 mL), and titrating with 0.025*N* Na_2SO_3 solution to the starch-iodine end point. Add to the neutralized sample the volume of Na_2SO_3 solution determined by the above test, mix, and after 10 to 20 min check sample for residual chlorine.

3) Samples containing other toxic substances—Certain industrial wastes, for example, plating wastes, contain toxic metals. Such samples often require special study and treatment.

4) Samples supersaturated with DO—Samples containing more than 9 mg DO/L at 20°C may be encountered in cold waters or in water where photosynthesis occurs. To prevent loss of oxygen during incubation of such samples, reduce DO to saturation at 20°C by bringing sample to about 20°C in partially filled bottle while agitating by vigorous shaking or by aerating with compressed air.

5) Sample temperature adjustment—Bring samples to 20 ± 1°C before making dilutions.

6) Nitrification inhibition—If nitrification inhibition is desired add 3.33 mg 2-chloro-6 (trichloro methyl) pyridine to each bottle before capping or add sufficient amounts to the dilution water to make a final concentration of 10 mg/L. Samples that may require nitrification inhibition include, but are not limited to, biologically treated effluents, samples seeded with biologically treated effluents, and river waters. Note the use of nitrogen inhibition in reporting results.

f. Dilution technique: Dilutions that result in a residual DO of at least 1 mg/L and a DO uptake of at least 2 mg/L after 5 d incubation produce the most reliable results. Make several dilutions of prepared

sample to obtain DO uptake in this range. Experience with a particular sample will permit use of a smaller number of dilutions. A more rapid analysis, such as COD, may be correlated approximately with BOD and serve as a guide in selecting dilutions. In the absence of prior knowledge, use the following dilutions: 0.0 to 1.0% for strong industrial wastes, 1 to 5% for raw and settled wastewater, 5 to 25% for biologically treated effluent, and 25 to 100% for polluted river waters.

Prepare dilutions either in graduated cylinders and then transfer to BOD bottles or prepare directly in BOD bottles. Either dilution method can be combined with any DO measurement technique. The number of bottles to be prepared for each dilution depends on the DO technique and the number of replicates desired.

When using graduated cylinders to prepare dilutions, and when seeding is necessary, either add seed directly to dilution water or to individual cylinders before dilution. Seeding of individual cylinders avoids a declining ratio of seed to sample as increasing dilutions are made. When dilutions are prepared directly in BOD bottles and when seeding is necessary, add seed directly to dilution water.

1) Dilutions prepared in graduated cylinders—If the azide modification of the titrimetric iodometric method (Section 421B) is used, carefully siphon dilution water, seeded if necessary, into a 1- to 2-L-capacity graduated cylinder. Fill cylinder half full without entraining air. Add desired quantity of carefully mixed sample and dilute to appropriate level with dilution water. Mix well with a plunger-type mixing rod; avoiding entraining air. Siphon mixed dilution into two BOD bottles. Determine initial DO on one of these bottles. Stopper the second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, siphon dilution mixture into one BOD bottle. Determine initial DO on this bottle and replace any displaced contents with sample dilution to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20°C.

2) Dilutions prepared directly in BOD bottles—Using a wide-tip volumetric pipet, add the desired sample volume to individual BOD bottles of known capacity. Fill bottles with enough dilution water, seeded if necessary, so that insertion of stopper will displace all air, leaving no bubbles. For dilutions greater than 1:100 make a primary dilution in a graduated cylinder before making final dilution in the bottle. When using titrimetric iodometric methods for DO measurement, prepare two bottles at each dilution. Determine initial DO on one bottle. Stopper second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, prepare only one BOD bottle for each dilution. Determine initial DO on this bottle and replace any displaced contents with dilution water to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20°C.

g. Determination of initial DO: If the sample contains materials that react rapidly with DO, determine initial DO immediately after filling BOD bottle with diluted sample. If rapid initial DO uptake is insignificant, the time period between preparing dilution and measuring initial DO is not critical.

Use the azide modification of the iodometric method (Section 421B) or the membrane electrode method (Section 421F) to determine initial DO on all sample dilutions, dilution water blanks, and where appropriate, seed controls.

For activated sludge samples use either the membrane electrode method or the $CuSO_4$-sulfamic acid modification of the iodometric method (Section 421E). For muds use either the membane electrode method or the alum flocculation modification of the iodometric method (Section 421D).

h. Dilution water blank: Use a dilution

BOD-10

water blank as a rough check on the quality of unseeded dilution water and cleanliness of incubation bottles. Together with each batch of samples incubate a bottle of unseeded dilution water. Determine initial and final DO as in 5*g* and 5*j*. The DO uptake should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.

i. Incubation: Incubate at 20°C ± 1°C BOD bottles containing desired dilutions, seed controls, dilution water blanks, and glucose-glutamic acid checks. Water-seal bottles as described in 5*f*.

j. Determination of final DO: After 5 d incubation determine DO in sample dilutions, blanks, and checks as in 5*g*.

6. Calculation

When dilution water is not seeded:

$$\text{BOD, mg/L} = \frac{D_1 - D_2}{P}$$

When dilution water is seeded:

$$\text{BOD, mg/L} = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

where:

D_1 = DO of diluted sample immediately after preparation, mg/L,
D_2 = DO of diluted sample after 5 d incubation at 20°C, mg/L,
P = decimal volumetric fraction of sample used,
B_1 = DO of seed control before incubation, mg/L,
B_2 = DO of seed control after incubation, mg/L, and
f = ratio of seed in sample to seed in control = (% seed in D_1)/(% seed in B_1).

If more than one sample dilution meets the criteria of a residual DO of at least 1 mg/L and a DO depletion of at least 2 mg/L and there is no evidence of toxicity at higher sample concentrations or the existence of an obvious anomaly, average results in the acceptable range.

In these calculations, corrections are not made for DO uptake by the dilution water blank during incubation. This correction is unnecessary if dilution water meets the blank criteria stipulated above. If the dilution water does not meet these criteria, proper corrections are difficult and results become questionable.

7. Precision and Accuracy

In a series of interlaboratory studies,[2] each involving 86 to 102 laboratories (and as many river water and wastewater seeds), 5-d BOD measurements were made on synthetic water samples containing a 1:1 mixture of glucose and glutamic acid in the total concentration range of 5 to 340 mg/L. The regression equations for mean value, $\bar{X}$, and standard deviation, S, from these studies were:

$$\bar{X} = 0.665 \text{ (added level, mg/L)} - 0.149$$

$$S = 0.120 \text{ (added level, mg/L)} + 1.04$$

For the 300-mg/L mixed primary standard, the average 5-d BOD was 199.4 mg/L with a standard deviation of 37.0 mg/L.

8. References

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2. U.S. ENVIRONMENTAL PROTECTION AGENCY, OFFICE OF RESEARCH & DEVELOPMENT. 1978. Personal communication, D.W. Ballinger to G.N. McDermott. Environmental Monitoring & Support Lab., Cincinnati, Ohio.

9. Bibliography

THERIAULT, E.J., P.D. MCNAMEE & C.T. BUTTERFIELD. 1931. Selection of dilution water for use in oxygen demand tests. *Pub. Health Rep.* 46:1084.

LEA, W.L. & M.S. NICHOLS. 1937. Influence of phosphorus and nitrogen on biochemical oxygen demand. *Sewage Works J.* 9:34.

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SAWYER, C.N. & L. BRADNEY. 1946. Modernization of the BOD test for determining the efficiency of the sewage treatment process. *Sewage Works J.* 18:1113.

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508 OXYGEN DEMAND (CHEMICAL)*

The chemical oxygen demand (COD) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to BOD, organic carbon, or organic matter. The test is useful for monitoring and control after correlation has been established. The dichromate reflux method is preferred over procedures using other oxidants because of superior oxidizing ability, applicability to a wide variety of samples, and ease of manipulation. Oxidation of most organic compounds is 95 to 100% of the theoretical value. Pyridine and related compounds resist oxidation and volatile organic compounds are oxidized only to the extent that they remain in contact with the oxidant. Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentration of free chloride ions.

1. Selection of Method

The open reflux method (A) is suitable for a wide range of wastes where a large sample size is preferred. The closed reflux methods (B and C) are more economical in the use of metallic salt reagents, but require homogenization of samples containing suspended solids to obtain reproducible results. Ampules and culture tubes with premeasured reagents are available commercially. Follow instructions furnished by the manufacturer.

Determine COD values of > 50 mg O_2/L by using procedures 508A.4*a*, 508B.4, or 508C.4. Use procedure 508A.4*b* to determine, with lesser accuracy, COD values from 5 to 50 mg O_2/L.

2. Interferences and Limitations

Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when silver sulfate (Ag_2SO_4) is added as a catalyst. However, Ag_2SO_4 reacts with chloride, bromide, and iodide to produce precipitates that are oxidized only partially. The difficulties caused by the presence of the halides can be overcome largely, though not completely, by complexing with mercuric sulfate ($HgSO_4$) before the refluxing procedure. Although 1 g $HgSO_4$ is specified for 50 mL sample, a lesser amount may be used where sample chloride con-

*Approved by Standard Methods Committee, 1985.

5/022__-0-7/87 Oil & Grease (Grav.) (Water) 7/30/87

U.S. Environmental Protection Agency
HWI Sample Management Office
P.O. Box 818, Alexandria, Virginia 22313
Phone: (703) 557-2490 or FTS-557-2490

OG+1

SAS Number

Scheduled for Approval

Special Analytical Services
Regional Request

[X] Regional Transmittal

[ ] Telephone Request

A. EPA Region and Site Name: Region V
B. Regional Representative: Jan Pels
C. Telephone Number: ( ) (312) 353 2720
D. Data request:
E. Site Name: Himco Dump Elkhart IN

Please provide below a description of your request for Special Analytical Services under the Uncontrolled Hazardous Waste Dumpsite Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Determination of "Oil and Grease" (Gravimetric, Separatory Funnel Extraction) in water (groundwater, surface water, leachate, etc.). Results will be reported as mg/l.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

5 low level aqueous samples

3. Purpose of analysis (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.):

Superfund Remedial

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment: daily by overnight carrier

O&G-2

6. Approximate number of days results required after lab receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

EPA Method 413.1 (Gravimetric, Separatory Funnel Extraction), 1983 ed. Samples will be in 1 qt. or 1 liter glass bottles and preserved with 1 to 2 ml/l of H_2SO_4 to pH < 2. Sample holding time is 28 days from date of sample collection. Samples will be kept at 4°C until analysis. Sample volume is best calculated from weights of sample bottles when full and empty. Any duplicate samples wil be field duplicates.

8. Special technical instructionns (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Check sample pH (wide range pH paper). If pH > 2, contact CPMS, CRL for further instructions. A 90 ml solvent blank is necessary for each solvent lot (Section 8.1 of Method 413.1) will be done at least in duplicate, and will be subtracted from sample residue values. Each 30 ml solvent extract will be added to sample bottle (Section 7.4 of Method 413.1), sample bottle and cap extracted, and solvent added to separatory funnel. Use only the method specified. Matrix spikes and laboratory blanks will be prepared from tap water, H_2SO_4, and Wesson oil. If sample results are expected to be greater than 10,000 mg/l (visual observation), contact CPMS, CRL prior to analysis.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Bench records of tare weights, final weights, sample volumes or weights, matrix spike weights, solvent blank weights, lab blank weights, etc., will be provided along with copies of worksheets used to calculate results. In case narrative and on bench records identify any problem samples as to emulsion problems, interferences, etc. All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. Other (use additional sheets or attach supplementary information, as needed):

11. Name of sampling/shipping contact: Greg Ruechel

Phone: 414 458 8711

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please call the Sample Management Office.

O+G-3

I. DATA REQUIREMENTS

Parameter	Detection Limit	Precision Desired (±% or Conc.)
Oil and Grease (Grav.)	< 5 mg/l	Any designated field duplicate values should not exceed ± 25% or ± 4 mg/l.
NOTE: These are minimum requirements. Report the actual detection limits used.		

II. QUALITY CONTROL REQUIREMENTS

Audits Required	Frequency of Audits	Limits* (±% or Conc.)
Solvent Blank (90 ml of Freon)	twice per solvent lot and sample set	< 3 mg for average value and must not exceed 2 mg in difference of results.
Lab Blank (1 liter of tapwater)	at least 1 per group of 10 samples or less	-3 to +3 mg/l
Matrix spike (1 liter of tapwater plus 15-20 mg of Wesson Oil)	at least 1 per group of 10 samples or less	80 - 120 % Recovery

III. *Action Required if Limits are Exceeded:

Take corrective action - Contact Jay Thakkar (312) 886-1972 or Chuck Elly (312) 353-9087.

BROMIDE

Br-1

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Client Request

☐ Regional Transmittal ☐ Telephone Request

A. EPA Region/Client: V

B. RSCC Representative: Jan Pels

C. Telephone Number: (312) 353-2720

D. Date of Request:

E. Site Name: Himco Dump, Elkhart Indiana

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: Analysis of 21 low level groundwater samples for bromide. Samples will be filtered in the field through a 0.45 micron filter and are to be analyzed within 48 hours of collection, not receipt.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):

21 whole aqueous samples, low level (historical levels 0.7 - 7 mg/L)

3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.):

Superfund remedial investigation

Br-2

4. Estimated date(s) of collection: August 1991

5. Estimated date(s) and method of shipment: daily by overnight carrier, no samples will be shipped Fridays for Saturday delivery

6. Number of days analysis and data required after laboratory receipt of samples: 30

7. Analytical protocol required (attach copy if other than a protocol currently used in this program): USGS method I-2057-85 "Anions, Ion-Exchange chromatographic, automated" analyze within 48 hrs of collection

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): optimize ion chromatographic conditions to separate Bromide from nitrate + sulfate as only bromide is to be analyzed + reported using this SAS. Select separator + suppressor columns for optimum bromide resolution. Calibration curve should include at least 5 points between

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. Report results as mg/L dissolved bromide. Bench records tabulating the order + preparation of calibration standards, calibration Verification Standards, cal curves, lab control standards, lab blanks, lab duplicates, lab matrix spikes

10. Other (use additional sheets or attach supplementary information, as needed): See Attachment 10

11. Name of sampling/shipping contact: Greg Ruechel
Phone: (414) 458-8711

with associated chromatograms + concentrations reported by the integrator. A photocopy of the instrument readouts and the settings (conductivity ion chromatograph operating parameters) should be included. All records of analysis must be legible and sufficient to calculate all concentrations and final results.

APPENDIX B

DQO SUMMARY SHEETS

DQO SUMMARY FORM

1. SITE

NAME Himco Dump
LOCATION Elkhart Indiana
NUMBER IND 980500292

EPA REGION V
PHASE II
RI1 (RI2) RI3 ERA FS RD RA (CIRCLE ONE)

2. MEDIA (CIRCLE ONE): SOIL | GW | SW/SED | AIR | BIO | (OTHER LEACHATE)

3. USE (CIRCLE ALL THAT APPLY): (SITE CHARAC. (H&S)) | RISK ASSESS. | (EVAL. ALTS.) | ENGG DESIGN | PRP DETER. | MONITORING REMEDIAL ACTION | OTHER

4. OBJECTIVE Characterize leachate quality to assess remedial alternatives that are feasible to pump + treat leachate

5. SITE INFORMATION

AREA ~40 acres DEPTH TO GROUND WATER 10-15'
GROUND WATER USE shallow aquifer wells abandoned; deep aquifer not used downgradient of site for residences (on city-supplied water)
SOIL TYPES CaSO4, landfill cap, loamy coarse sand + gravel
SENSITIVE RECEPTORS wetland, residences immediately downgradient

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA
(D.O.) (pH) (CONDUCTIVITY) (VOA) (ABN) TCLP
(PESTICIDES) PCB (METALS) (CYANIDE) (Bromide)
TOX TOC BTX COD alkalinity
(BOD) (O+G) (Total Phenol) VSS (NH3) ISS TKN TDS (NO2+NO3) Cl SO4 TP

B. PHYSICAL DATA
PERMEABILITY HYDRAULIC HEAD
POROSITY PENETRATION TEST
GRAIN SIZE HARDNESS
BULK DENSITY

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL | BIASED | (GRAB) | NON-INTRUSIVE | PHASED
SOURCE | GRID | COMPOSITE | (INTRUSIVE) – FROM TEST PITS

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT HNu, CH_4, H_2S, pH, conductivity
~~LEVEL 2 FIELD ANALYSIS - EQUIPMENT~~ Dissolved oxygen
LEVEL 3 NON-CLP LABORATORY - METHODS
LEVEL 4 CLP/RAS - METHODS VOA, BNA, METALS/CN, Pesticides/PCBs
LEVEL 5 NON STANDARD Br, COD, alkalinity, BOD, O+G, Total phenol, VSS, TSS, TDS, Cl, SO_4, NH_3, NO_2+NO_3, TKN, TP

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR not applicable
CRITICAL (LIST) 1 leachate in PNA area, 1 leachate in fill area
PROCEDURES test pit/trench dug with backhoe, sample collected from liquid that oozes into trench

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD
COLLOCATED - 5% OR 0
REPLICATE - 5% OR 1
FIELD BLANK - 5% OR 1
TRIP BLANK - 1 PER DAY OR 1*

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR ≥ 5%
REPLICATE - 1 PER ANALYSIS BATCH OR ↓
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR ↓
OTHER

*assumes all 3 trenches sampled on same day in one cooler

11. BUDGET REQUIREMENTS

BUDGET Level B SCHEDULE JULY-AUGUST 1991
STAFF backhoe operator, geologist, H+S officer

CONTRACTOR Donohue PRIME CONTRACTOR Mathes
SITE MANAGER Vanessa Harris DATE JUNE 1991

1. SITE

NAME Hmco Dump
LOCATION Elkhart IN
NUMBER IND 980500 292

EPA REGION V
PHASE II
RI 1 (RI 2) RI 3 ERA FS RD RA (CIRCLE ONE)

2. MEDIA (CIRCLE ONE)	SOIL	GW	(SW/SED)	AIR	(BIO Benthos ID)	OTHER

3. USE (CIRCLE ALL THAT APPLY)	(SITE CHARAC. (H&S))	(RISK ASSESS.)	(EVAL ALTS.)	ENGG DESIGN	PRP DETER.	MONITORING REMEDIAL ACTION	OTHER

4. OBJECTIVE assess if surface water bodies impacted at depths where biota (fish) reside; determine if AWQC exceedances of Pb & Cr exist at depth; ID benthos to check for species indicative of stress; measure organic layer to provide volume for any remediation.

5. SITE INFORMATION (see other sheets)

AREA _____ DEPTH TO GROUND WATER _____
GROUND WATER USE _____
SOIL TYPES _____
SENSITIVE RECEPTORS _____

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA

(D.O.) (pH) (CONDUCTIVITY) (VOA) (ABN) TCLP — (SW only)

(PESTICIDES) (PCB) (METALS) (CYANIDE)

TOX (TOC) (BTX) (COD) — (TOC, COD = sed only; surface water)

WATER QUALITY: Br, Cl, SO4, alkalinity, NH_3, NO_2 & NO_3, TKN, TP, TDS, TSS

B. PHYSICAL DATA

PERMEABILITY, POROSITY, GRAIN SIZE, BULK DENSITY

HYDRAULIC HEAD, PENETRATION TEST, HARDNESS

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL	BIASED	(GRAB)	NON-INTRUSIVE	PHASED
SOURCE	GRID	COMPOSITE	(INTRUSIVE)	

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT D.O., pH, conductivity, depth of organic layer
LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____
LEVEL 3 NON-CLP LABORATORY - METHODS _____
LEVEL 4 CLP/RAS - METHODS VOA, ABN, PCB/pesticides, Metals/CN.
LEVEL 5 NON STANDARD TOC, Water quality

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR 3 sw/sed from 1 upgradient surface water body
CRITICAL (LIST) all
PROCEDURES dredge + gravity corer, remmerer bottle

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD
COLLOCATED - 5% OR _____
REPLICATE - 5% OR 10%
FIELD BLANK - 5% OR 10% (surface water)
TRIP BLANK - 1 PER DAY OR ~~10%~~ 1 (surface water)

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR 5%
REPLICATE - 1 PER ANALYSIS BATCH OR "
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR "
OTHER _____

11. BUDGET REQUIREMENTS

BUDGET _____ SCHEDULE July 1991 - Aug 1991
STAFF 2 samples collected with fish

CONTRACTOR Donohue PRIME CONTRACTOR Life Systems - ID benthos
SITE MANAGER Varena Harris DATE June 1991

FOR DETAILS SEE SAMPLING & ANALYSIS PLAN

COM SF 000 1.002

1. SITE

NAME Homco Dump
LOCATION Elkhart IN
NUMBER IND 980500292

EPA REGION V
PHASE II
RI1 (RI2) RI3 ERA FS RD RA
(CIRCLE ONE)

2. MEDIA (CIRCLE ONE)	(SOIL)	GW	SW/SED	AIR	BIO	OTHER

3. USE (CIRCLE ALL THAT APPLY)	(SITE CHARAC. (H&S))	(RISK ASSESS.)	EVAL. ALTS.	ENGG DESIGN	PRP DETER.	MONITORING REMEDIAL ACTION	OTHER

4. OBJECTIVE assess if bike trail soil presents risk to dirt bike riders; determine if site runoff has occurred to west of surface water bodies; assess if site wetland soil impacted by leachate/CaSO4.

5. SITE INFORMATION

AREA _____ DEPTH TO GROUND WATER _____
GROUND WATER USE _____
SOIL TYPES see other sheets
SENSITIVE RECEPTORS _____

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA

pH	(PESTICIDES)	TOX	
CONDUCTIVITY	PCB	(TOC)	
(VOA)	METALS	BTX	
(ABN)	(CYANIDE)	COD	
TCLP			

B. PHYSICAL DATA

PERMEABILITY	HYDRAULIC HEAD
POROSITY	PENETRATION TEST
(GRAIN SIZE)	HARDNESS
BULK DENSITY	
(depth of organic layer)	

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL SOURCE	(BIASED) GRID	(GRAB) COMPOSITE	NON-INTRUSIVE (INTRUSIVE - 0 - 18")	PHASED

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT measure depth of organic layer
LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____
LEVEL 3 NON-CLP LABORATORY - METHODS _____
LEVEL 4 CLP/RAS - METHODS VOA, BNA, PCB/pesticide, metals/CN
LEVEL 5 NON STANDARD TOC, grain size, SAS methods

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR None
CRITICAL (LIST) all
PROCEDURES hand dug

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD
COLLOCATED - 5% OR 10% min
REPLICATE - 5% OR 10%
FIELD BLANK - 5% OR 0
TRIP BLANK - 1 PER DAY OR 0

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR 5%
REPLICATE - 1 PER ANALYSIS BATCH OR ↓
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR ↓
OTHER _____

11. BUDGET REQUIREMENTS

BUDGET _____ SCHEDULE July-Aug 1991
STAFF 2 field techs - to include wetland specialist +/or geologist

CONTRACTOR Donohue PRIME CONTRACTOR _____
SITE MANAGER Vanessa Harris DATE June 1991

FOR DETAILS SEE SAMPLING & ANALYSIS PLAN

COM SF 000 1.002

1. SITE

NAME Homco Dump
LOCATION Elkhart IN
NUMBER

EPA REGION V
PHASE II
RI 1 (RI 2) RI 3 ERA FS RD RA
(CIRCLE ONE)

2. MEDIA (CIRCLE ONE): (SOIL landfill cap) | GW | SW/SED | AIR | BIO | OTHER

3. USE (CIRCLE ALL THAT APPLY): SITE CHARAC. (H&S) | RISK ASSESS. | EVAL. ALTS. | (ENGG DESIGN) | PRP DETER. | MONITORING REMEDIAL ACTION | OTHER

4. OBJECTIVE determine if existing cap can support an additional cap

5. SITE INFORMATION

AREA ~40 acres
DEPTH TO GROUND WATER 10-15'
GROUND WATER USE shallow aquifer + deep aquifer not in use for drinking.
SOIL TYPES CaSO4 - cap cover; loamy coarse sand + gravel
SENSITIVE RECEPTORS wetland; residents downgradient

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA

pH	PESTICIDES	TOX
CONDUCTIVITY	PCB	TOC
VOA	METALS	BTX
ABN	CYANIDE	COD
TCLP		

B. PHYSICAL DATA

PERMEABILITY	HYDRAULIC HEAD
POROSITY	PENETRATION TEST
GRAIN SIZE	HARDNESS
BULK DENSITY	
triaxial compression	

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL (BIASED) GRID (GRAB) COMPOSITE | NON-INTRUSIVE (INTRUSIVE) | PHASED
SOURCE

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT
LEVEL 2 FIELD ANALYSIS - EQUIPMENT
LEVEL 3 NON-CLP LABORATORY - METHODS
LEVEL 4 CLP/RAS - METHODS
LEVEL 5 NON STANDARD ASTM method for triaxial compression

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR none
CRITICAL (LIST) all five locations
PROCEDURES

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD
COLLOCATED - 5% OR none
REPLICATE - 5% OR
FIELD BLANK - 5% OR
TRIP BLANK - 1 PER DAY OR

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR 0
REPLICATE - 1 PER ANALYSIS BATCH OR
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR
OTHER

11. BUDGET REQUIREMENTS

BUDGET
SCHEDULE July 91 - Aug 91
STAFF geotech engineer

CONTRACTOR Donohue
PRIME CONTRACTOR
SITE MANAGER Vanessa Harris
DATE June 1991

FOR DETAILS SEE SAMPLING & ANALYSIS PLAN

COM SF 000 1.002

APPENDIX C

SAMPLE BOTTLE CLEANING PROTOCOLS

Appendix C of the approved Final QAPP,
dated June 1990, applies and is not resubmitted

APPENDIX D

FIELD METER CALIBRATION PROCEDURES

HNU Model PI 101
Fisher Accumet Model 955
Cole Parmer Model 4070 Conductivity Meter
YSI Model 54A Dissolved Oxygen Meter
Lumidor Gasponder IV Model PGM-14

Appendix D of the approved Final QAPP, dated June 1990, applies and is not resubmitted

APPENDIX E

STANDARD OPERATING PROCEDURES
FOR
FIELD MEASUREMENTS

Volatile Organics by HNU

pH

Conductivity

Dissolved Oxygen

Methane and Hydrogen Sulfide

Appendix E of the approved Final QAPP,
dated June 1990, applies and is not resubmitted